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UltiMate 3000 Series

Photodiode Array Detector

Operating Instructions

UltiMate 3000 Photodiode Array Detector
RUN
DEUTERIUM
TUNGSTEN
POWER
DIONEX

Revision: 1.1
Date: October 2007

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DIONEX

CE

Declaration of Conformity

Product: Detector
Type: **PDA-3000**

Dionex Softron GmbH herewith declares conformity of the above products with the respective requirements of the following regulations:

- Low-Voltage Equipment Directive 73/23/EEC
  changed by 93/68/EEC
- EMC Directive 2004/108/EG

The electrical safety of the products was evaluated based on the following standard:

- EN 61010-1: 2002
  Safety requirements for electrical equipment for measurement, control and laboratory use
  Part 1: General Requirements

The Electromagnetic Compatibility (EMC) of the products was evaluated based on the following standard:

- DIN EN 61326: 2004
  Electrical equipment for measurement, control and laboratory use
  EMC Requirements

This declaration is issued for the manufacturer

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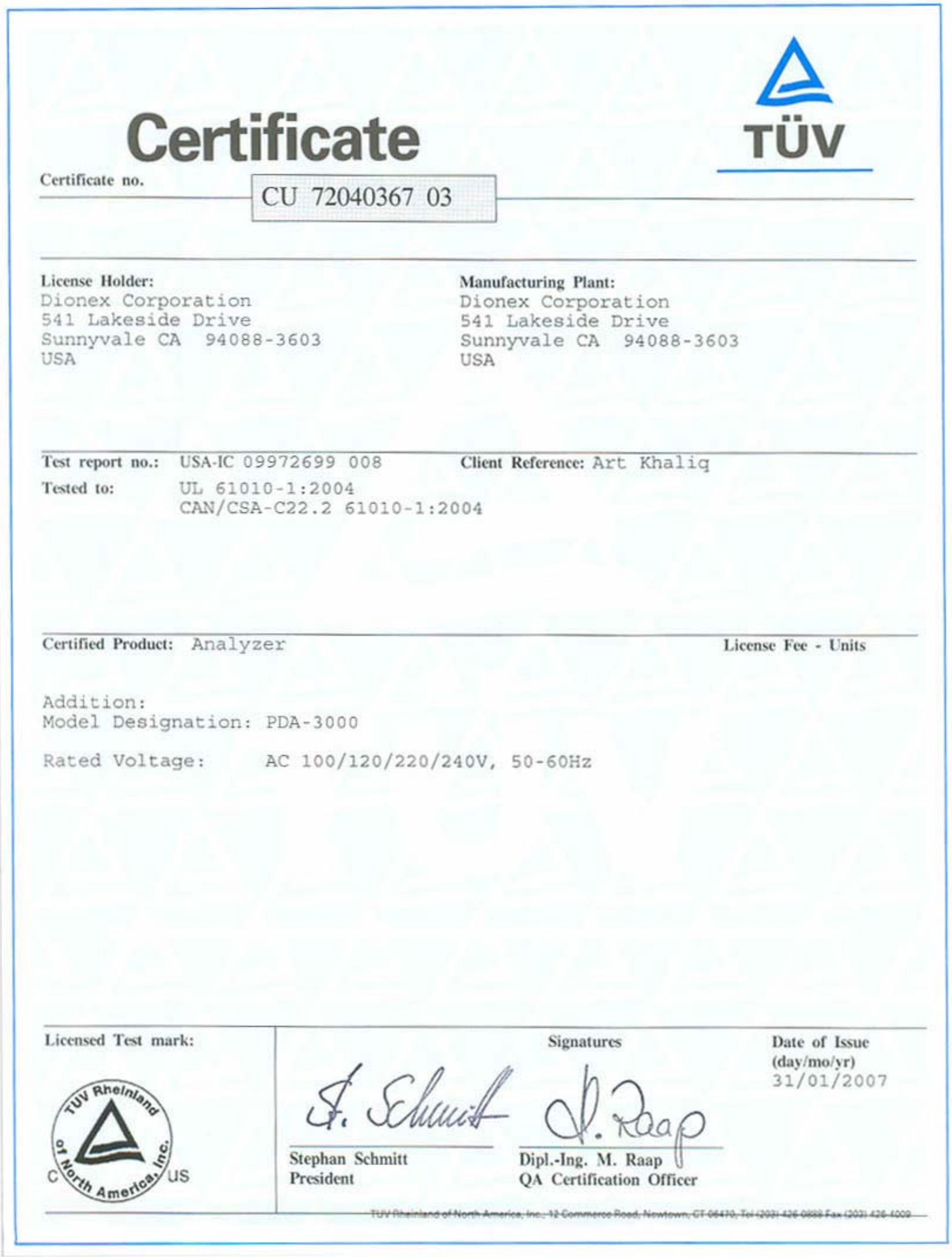
by the President, Dr. Peter Jochum.

August 1, 2007

Dr. Peter Jochum



Certificate

TÜV

Certificate no. CU 72040367 03

License Holder:	**Manufacturing Plant:**
Dionex Corporation 541 Lakeside Drive Sunnyvale CA 94088-3603 USA	Dionex Corporation 541 Lakeside Drive Sunnyvale CA 94088-3603 USA

Test report no.: USA-IC 09972699 008 **Client Reference:** Art Khaliq

Tested to:
UL 61010-1:2004
CAN/CSA-C22.2 61010-1:2004

Certified Product: Analyzer **License Fee - Units**

Addition:
Model Designation: PDA-3000

Rated Voltage: AC 100/120/220/240V, 50-60Hz

Licensed Test mark:

Signatures

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Date of Issue
(day/mo/yr)
31/01/2007

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Table of Contents

1 Introduction 1

1.1 How to Use This Manual 1
1.2 Safety Information 2
1.2.1 Symbols on the Instrument and in the Manual 2
1.2.2 General Safety Precautions 3
1.2.3 Consignes Générales de Sécurité 5
1.3 Product Overview 7
1.4 Theory of Photodiode Array Detection 8

2 Description 11

2.1 Front Panel 11
2.2 Inside Front Panel 13
2.3 Optical System 14
2.4 Flow Cells 15
2.5 Rear Panel 19
2.6 Leak Sensor 20
2.7 Mobile Phases 21
2.8 Solvent Delivery System 21

3 Installation 23

3.1 Facilities Required 23
3.2 Unpacking the PDA-3000 23
3.3 Positioning the Detector in the UltiMate 3000 System 24
3.4 Installing Chromeleon Software 26
3.5 Connecting the Detector 26
3.5.1 USB Connection 26
3.5.2 Analog Output Connection 27
3.5.3 Relay/TTL Connections 28
3.5.4 Connecting the Power Cord 28
3.6 Setting Up the Detector in Chromeleon 29
3.6.1 Loading the USB Driver for the Detector 29
3.6.2 Installing the Detector 30
3.7 Installing the Flow Cell 31
3.8 Connecting the Waste Tubing 33

4 Detector Control 35

4.1 Getting Started 35
4.2 Routine Operation by Chromeleon 35
4.2.1 Direct Control 36
4.2.2 Automated Control 37
4.2.3 Control Commands 42

4.3 Optimizing Detector Performance 44
  4.3.1 Flow Cell Material 45
  4.3.2 Rise Time 45
  4.3.3 Data Collection Rate 46
  4.3.4 Sample Wavelength 47
  4.3.5 Bandwidth 48
  4.3.6 Reference Wavelength 49
  4.3.7 Reference Bandwidth 50
  4.3.8 Bunch Width 52
  4.3.9 Step 52
  4.3.10 Average 53
  4.3.11 Negative Absorbance 54
4.4 Wellness and Diagnostics 55
4.5 Stand-Alone Analog Operation 58

5 Troubleshooting 59

5.1 Alarms and Error Conditions 60
5.2 Flashing LED 69
5.3 Lamp Does Not Light or Lamp LED Is Flashing 70
5.4 No Detector Response 70
5.5 Noisy Baseline 71
5.6 Drifting Baseline 74
5.7 Deuterium Lamp Intensity Reading Too Low 75
5.8 Wavelength Calibration Fails 76
5.9 Wavelength Verification Fails 78
5.10 No Spectra Collected 78
5.11 Low Spectral Resolution 79
5.12 Peaks Too Large or Small 79
5.13 Poor Peak Shape 80
5.14 Faulty USB Communication 81

6 Routine Maintenance 83

6.1 Regular Maintenance Procedures 83
6.2 Flow Cell 84
  6.2.1 Liquid Leaks 85
  6.2.2 Cleaning the Flow Cell 86
  6.2.3 Removing Trapped Air from the Flow Cell 86
  6.2.4 Replacing the Flow Cell 87
  6.2.5 Replacing the Flow Cell Windows and Seals 89
  6.2.6 Checking the Flow Cell for Leakage 91
6.3 Lamps 91
  6.3.1 Lamp Diagnostics 91
  6.3.2 Replacing the Deuterium Lamp 92
  6.3.3 Replacing the Tungsten Lamp 95
6.4 Replacing the Main Power Fuses 97

7 Specifications 99

8 Accessories, Spare Parts and Consumables 101

8.1.1 Standard Accessories (included in the Ship Kit) 101
8.1.2 Flow Cells 101
8.1.3 Spare Parts and Consumables 102

9 Technical Appendix - Relay/TTL Pin Assignment 103

10 Index 105

1 Introduction

1.1 How to Use This Manual

The layout of this manual is designed to provide quick reference to the sections of interest to the user. However, in order to obtain a full understanding of the detector, Dionex recommends that you review the manual thoroughly before beginning operation.

NOTE: The descriptions in this manual refer to firmware version 2.0 and Chromeleon 6.80 Service Pack 2.

The term "the detector" or "the PDA" is used throughout the manual.

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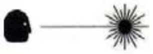
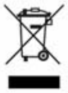
1.2 Safety Information

The CE Mark label and the cTUVus Mark safety label on the instrument indicate that the instrument is in compliance with the related standards (→ pages I and II).

1.2.1 Symbols on the Instrument and in the Manual

The table below shows the symbols used on the instrument:

Symbol	**Description**
~	Alternating current—Courant alternatif
|	Power supply is on—L'instrument est mis en circuit.
○	Power supply is off—L'instrument est mis hors circuit.
⚠	Refer to the operating instructions to prevent risk of harm to the operator and to protect the instrument against damage. Référez-vous au ce manuel pour éviter un risque de blessure à l'opérateur et/ou de protéger l'instrument contre des dommages.
	Harmful radiation—Avoid looking directly into light sources. Rayons dangereux—Ne regardez pas directement la lumière.
	WEEE (Waste Electrical and Electronic Equipment) label—For more information, refer to the WEEE Information section in the manual binder. Étiquette de WEEE (Waste Electrical and Electronic Equipment) —Pour plus d'information, référez-vous à la section WEEE Information dans ce fichier.

At various points throughout the manual, messages of particular importance are indicated by certain symbols:

⚠ **Danger**:	Indicates an imminently hazardous situation which, if not avoided, will result in death or serious injury.
⚠ **Warning**:	Indicates a potentially hazardous situation which, if not avoided, may result in death or serious injury.
⚠ **Caution**:	Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury.
Important:	Indicates that the function or process of the instrument may be impaired. Operation does not constitute a hazard.

Messages d’avertissement en français

⚠ **Danger**: Signale une situation de danger immédiat qui, si elle n'est pas évitée, entraînera des blessures graves à mortelles.

⚠ **Avertissement**: Signale une situation de danger potentiel qui, si elle n'est pas ´evitée, pourrait entra”ner des blessures graves à mortelles.

⚠ **Mise en garde**: Signale une situation de danger potentiel qui, si elle n'est pas évitée, pourrait entraîner des blessures mineures à modérées. Également utilisé pour signaler une situation ou une pratique qui pourrait gravement endommager l'instrument mais qui n'entra”nera pas de blessures.

Informational messages also appear throughout this manual. These are labeled NOTE. Notes call attention to certain information. They alert users to an unexpected result of an action, suggest how to optimize instrument performance, etc.

1.2.2 General Safety Precautions

When working with analytical instrumentation, you should know the potential hazards of using chemical solvents.

NOTE: Before initial operation of the detector, make sure that you are familiar with the contents of this manual.

Observe any warning labels on the device and refer to the related sections in these operating instructions.

For the general safety precautions in French, refer to Consignes Générales de Sécurité (→ page 5).

To avoid the possibility of personal injury and/or damage to the instrument, observe the following general safety precautions when operating the detector or carrying out any maintenance work:

Please observe the following general safety precautions when operating the instrument or carrying out any maintenance work:

- Install the HPLC system in a well-ventilated laboratory. If the mobile phase includes volatile or flammable solvents, do not allow them to enter the workspace.
- For minimum interference effects, all components of the analytical system should be connected to the same mains output (same phase).
- The front panel door tilts downward. To prevent damage to the detector when lifting or moving, always lift only from the bottom or by the sides. The open front panel door is not designed to carry weight. Therefore, you should not place any objects on the open door.
- After operation, rinse out buffers and solutions that form peroxides.

- If the mobile phase includes volatile or flammable solvents, avoid open flames and sparks.
- If a leak occurs, turn off the instrument and remedy the situation immediately.
- When the panels are removed, dangerous electrical connections will be exposed. Disconnect the detector from all power sources before removing the panels. Always replace blown fuses with original spare part fuses from Dionex (→ section 6.4, page 97).
- Replace faulty power cords and communication cables.
- Many organic solvents and buffers are toxic. Know the toxicological properties of all mobile phases that you are using.
- The toxicological properties of many samples may not be well known. If you have any doubt about a sample, treat it as if it contains a potentially harmful substance.
- Wear goggles when handling mobile phases or operating the instrument. An eye wash facility and a sink should be close to the unit. If any mobile phase splashes on the eyes or skin, wash the affected area and seek medical attention.
- Dispose of all waste mobile phase in an environmentally safe manner that is consistent with all local regulations. Do not allow flammable and/or toxic solvents to accumulate. Follow a regulated, approved waste disposal program. Never dispose of flammable and/or toxic solvents through the municipal sewage system
- Use only standard solvents (HPLC grade) and buffers that are compatible with all parts that may be exposed to solvents.
- In an UltiMate 3000 system, some components are made of PEEK. While this polymer has superb chemical resistance to most organic solvents, it tends to swell when in contact with trichlormethane ($CHCl_3$), dimethyl sulfoxide (DMSO), or tetrahydrofuran (THF). In addition, it is attacked by concentrated acids, such as, sulfuric acid and nitric acid or a mixture of hexane, ethyl actate, and methanol. (Swelling or attack by concentrated acids is not a problem with brief flushing procedures.)
- Do not use PEEK tubing that is stressed, bent, or kinked.
- During operation, the lamp and the surrounding parts become extremely hot and remain so for some time after the detector is turned off. To avoid possible injury, allow sufficient time for the lamp to cool before performing any maintenance or repair work.
- The deuterium lamp emits UV radiation that is harmful to the eyes. Therefore, avoid looking directly into the light source. Always turn off the detector before exchanging the lamp.
- Use original Dionex spare parts only. Substituting non-Dionex parts or using non-Dionex accessories may impair the performance of the instrument.
- Do not use the detector in ways other than those described in this manual.

1.2.3 Consignes Générales de Sécurité

Veuillez noter: Avant de commencer à utiliser l'instrument, assurez-vous que vous vous êtes familiarisés avec le contenu de ce manuel.

Observez les étiquettes d'avertissement sur l'appareil et référez-vous aux sections correspondantes dans ce mode d'emploi.

Veuillez observer les consignes générales de sécurité suivantes lorsque vous utilisez l'instrument ou que vous procédez à des opérations de maintenance:

- Installez le système HPLC dans un laboratoire bien ventilé. Si la phase mobile contient des solvants volatils ou inflammables, empêchez qu'ils ne pénètrent dans l'espace de travail.
- Afin d'éviter au maximum les interférences, tous les éléments du système analytique doivent être raccordés à la même ligne secteur (même phase).
- Lorsque vous soulevez ou l’instrument, tenez-le toujours par le dessous ou par les côtés de l'unité. Soulever l’instrument par la partie avant inférieure ou par le panneau avant peut endommager la porte.
- Ne placez aucun objet lourd sur la porte ouverte du panneau avant. Ceci pourrait endommager la porte.
- Après utilisation, purgez le système des tampons et des susceptibles de former des peroxydes.
- Si la phase mobile contient des solvants volatils ou inflammables, évitez les flammes nues et les sources d’étincelles à proximité.
- Si une fuite se produit, arrêtez immédiatement l’instrument et remédiez au problème.
- Quand les capots sont démontés, des connexions électriques sous haute tension deviennent accessibles. Débranchez l'instrument de toute source d'alimentation électrique avant de retirer les capots.
- Remplacez toujours les fusibles grillés par des fusibles de rechange d'origine Dionex. (→ chapitre 0 (→ page 95).
- Remplacez les cordons d'alimentation électrique et les câbles de communication défectueux.
- De nombreux solvants organiques et solutions salines sont toxiques. Informez-vous des propriétés toxicologiques de toutes les phases mobiles que vous utilisez.
- Les propriétés toxicologiques de nombreux échantillons peuvent être mal connues. Au moindre doute concernant un échantillon, traitez-le comme s'il contenait une substance potentiellement dangereuse.

- Portez des lunettes de protection lorsque vous manipulez des phases mobiles ou que vous utilisez l'instrument. Une installation permettant de se laver les yeux ainsi qu'un lavabo doivent se trouver à proximité du système. Si une phase mobile, quelle qu'elle soit, gicle dans les yeux ou sur la peau, lavez la zone affectée et consultez un médecin.
- Débarrassez-vous de tous les déchets de phase mobile de manière écologique, conformément à la règlementation en vigueur au niveau local. Empêchez impérativement l'accumulation de solvants inflammables et/ou toxiques. Suivez un programme d'élimination des déchets règlementé et approuvé. Ne jetez jamais de solvants inflammables et/ou toxiques dans le système municipal d'évacuation des eaux usées.
- Utilisez uniquement des solvants (qualité HPLC) et des solutions salines compatibles avec les matériaux exposés phase mobiles.
- Dans un système UltiMate 3000, certaines composantes sont en PEEK. Bien que ce polymère présente une excellente résistance chimique à la plupart des solvants organiques, il a tendance à gonfler lorsqu'il est en contact prolongé avec du chloroforme ($CHCl_3$), du diméthyle sulfoxyde (DMSO) ou du tétrahydrofurane (THF). De plus, il est attaqué par des acides concentrés tels que l'acide sulfurique et l'acide nitrique ou d'un composé du hexane, éthyle acétate et méthanol. (Ces acides peuvent cependant être utilisés dans le cadre de procédures de nettoyage, à condition que l’exposition soit brève.)
- N'utilisez pas de tubes PEEK écrasés, pliés ou abimés.
- Au cours du fonctionnement, la lampe et les pièces qui l'entourent deviennent extrêmement chaudes et le restent pendant quelques temps après que le détecteur ait été arrêté. Afin d'éviter de possibles brûlures, laissez la lampe refroidir pendant une période suffisamment longue avant de procéder à tous travaux de maintenance ou de réparation
- La lampe au deutérium émet des rayons UV qui sont dangereux pour les yeux. Par conséquent, évitez de regarder directement la source de lumière et portez des lunettes de protection. Arrêtez toujours le détecteur avant de remplacer la lampe
- Utilisez des pièces de rechange d'origine Dionex. Effectuer des remplacements par des pièces ne provenant pas de Dionex ou utiliser des accessoires ne provenant pas de Dionex peut affecter les performances de l'instrument.
- N'utilisez pas l'instrument de manière autre que celles décrites dans ce manuel.

1.3 Product Overview

The Dionex PDA-3000 Photodiode Array Detector is an optical detector capable of measuring the absorbance spectrum from 190 to 800 nm. A deuterium lamp optimizes the UV range (190 to 380 nm) and a tungsten lamp optimizes the visible range (380 to 800 nm).

When using the PDA-3000, up to five single wavelengths (2D chromatograms) can be collected without being required to collect 3D data. Collecting individual wavelengths instead of the spectra offers two advantages:

- It eliminates the need to perform extractions for analyses that do not require spectral data
- It conserves disk space.

Key features of the PDA-3000 include:

- The deuterium and tungsten lamps ensure low baseline noise, high signal intensity, and applications flexibility in the wavelength range from 190 to 800 nm.
- A 1024-element photodiode array optimizes spectral resolution.
- A built-in holmium oxide filter verifies wavelength calibration accuracy.
- Five flow cells are available for analytical, semi-micro, and semipreparative applications.
- The front panel LEDs indicate detector status at a glance.
- Four analog outputs support data collection on alternate data collection systems.

The PDA-3000 communicates with the computer through USB (Universal Serial Bus). It is controlled remotely by a computer running the Chromeleon® Chromatography Management System. However, it can also be operated with other data systems, such as Analyst® (Applied Biosystems/MDS Sciex), HyStar™ (Bruker Daltonics), or Xcalibur™ (Thermo Scientific). To do so, installation of the DCMS$^{\text{Link}}$ (Dionex Chromatography Mass Spectrometry Link) software is required.

To take full advantage of the capabilities of the PDA-3000, order Chromeleon with the PDA Control option. This option is required in order to perform:

- 3D data presentation and spectral analysis (real-time or post-run)
- Peak purity analysis with selectable criteria
- Spectral library search for positive peak identification

1.4 Theory of Photodiode Array Detection

Monochromatic variable wavelength detectors monitor eluting components of the sample at a single wavelength (ideally, the wavelength of maximum absorbance). Photodiode array (PDA) detectors scan a range of wavelengths every few milliseconds and continually generate spectral information. Wavelength, time, and absorbance can all be plotted.

For example, Fig. 1 (→ page 8) shows an overlay of pyrene and an impurity obtained from two consecutive chromatographic analyses. Based on 2D data, or if the analyses had been performed with a conventional variable wavelength detector, these peaks might have been misidentified as the same component. A PDA-3000, under the control of Chromeleon with the PDA Control option, correctly identified and differentiated the components. The spectra of pyrene and the impurity clearly indicate that the two peaks are different components (→ Fig. 2, page 9).

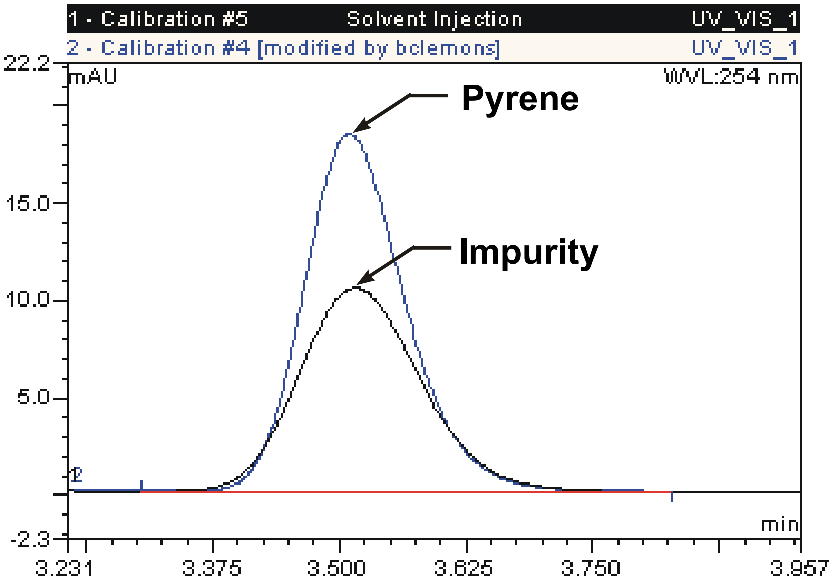


Fig. 1: Overlay of Pyrene and an Impurity

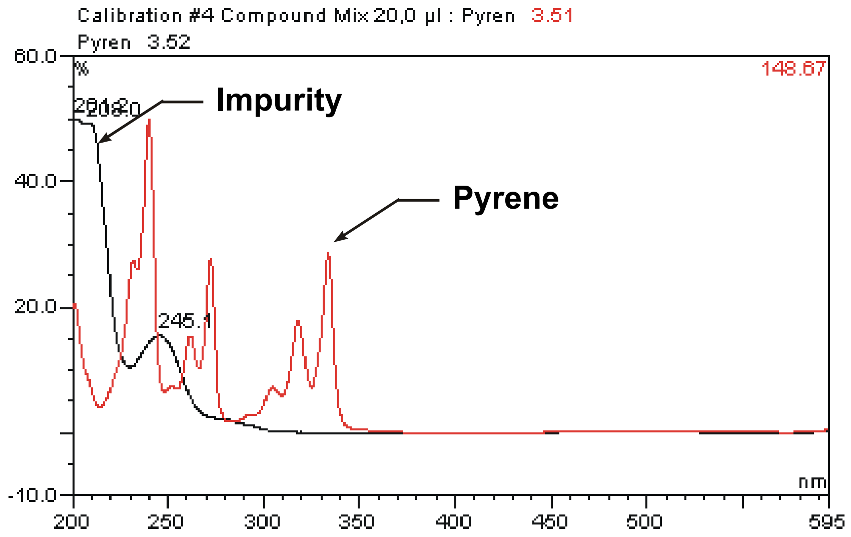


Fig. 2: Spectra of Pyrene and an Impurity

PDA detectors provide three-dimensional information that allows an accurate assessment of peak identity, purity, and quantitation in a single analysis. Software support for PDA detectors includes peak purity and spectral library search functions to help determine peak homogeneity and identity.

2 Description

2.1 Front Panel

Fig. 3 illustrates the PDA-3000 Photodiode Array Detector front panel.

- Press the Power switch in the lower left corner for on/off control of the detector power.
- The four LED indicators identify the status of the detector power, the deuterium and tungsten lamps, and the analysis (see table below).

When the corresponding function is in progress, the LED is lighted. If an error occurs, the LED flashes and an error message is logged in the Chromeleon Audit Trail.

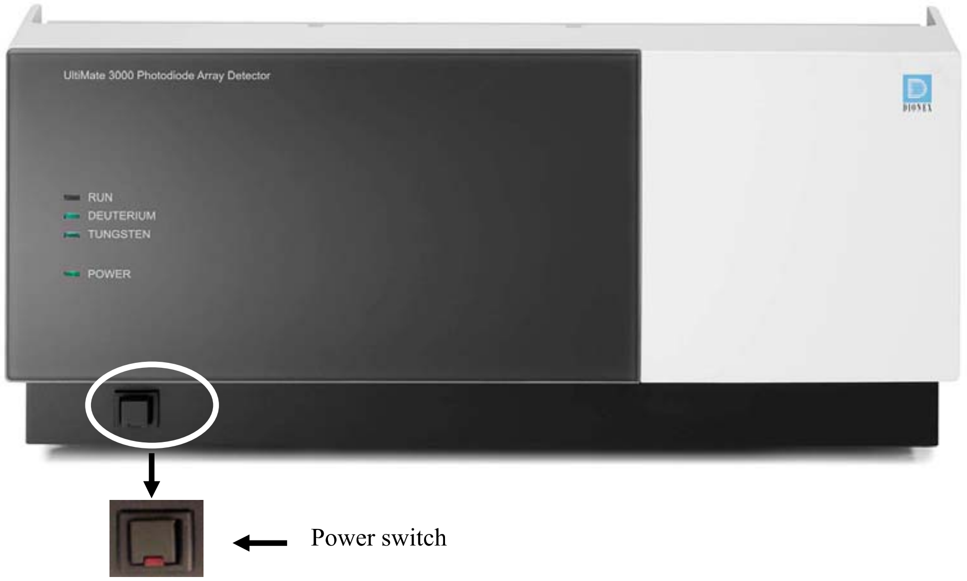


Fig. 3: PDA-3000 Photodiode Array Detector

LED	LED Status	Function
Run	Lighted	Data is being acquired by Chromeleon.
	Flashing	The leak sensor in the drip tray was triggered.
	Off	Data is not being acquired by Chromeleon.
Deuterium	Lighted	The deuterium lamp is on.
	Flashing	A command was issued to turn on the deuterium lamp, but the lamp failed to turn on (→ section 5.3, page 70) If a subsequent attempt to turn on the lamp is successful, the LED will automatically stop flashing.
	Off	The deuterium lamp is off.

LED	LED Status	Function
Tungsten	Lighted	The tungsten lamp is on.
	Flashing	A command was issued to turn on the tungsten lamp, but the lamp failed to turn on (→ section 5.3, page 70). If a subsequent attempt to turn on the lamp is successful, the LED will automatically stop flashing.
	Off	The tungsten lamp is off.
Power	Lighted	The detector power is on. Press the **Power** switch for on/off control of the detector.
	Flashing	The detector failed the start-up routine (→ section 5.2, page 69).
	Off	The detector power is off. Press the **Power** switch for on/off control of the detector.

Table: PDA-3000 Front Panel LEDs

2.2 Inside Front Panel

Grasp the PDA-3000 front panel by the sides and pull it straight off to access the inside front panel.

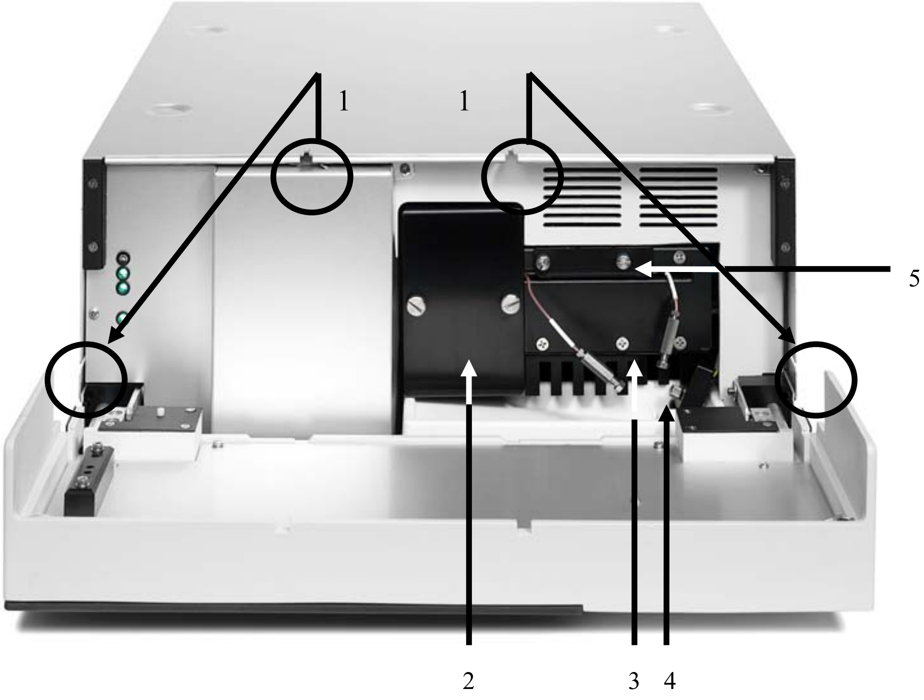


Fig. 4: PDA-3000 Inside Front Panel

No.	Description
1	Slots in the enclosure to thread the flow cell capillaries to the outside
2	Flow cell cover. The flow cell cover protects the cell from dust, ambient light, fluctuations in ambient temperature, and vibration. Note that the PDA-3000 is shipped without a flow cell. Flow cells must be ordered separately.
3	Lamp cover. The lamp cover enhances the thermal stabilization of the optical bench. The deuterium and/or tungsten lamp is installed behind the cover (→ page 91).
4	Leak sensor (→ page 20)
5	Tubing bracket The bracket holds the flow cell inlet tubing with the heat exchanger.

2.3 Optical System

Fig. 5 is a schematic of the PDA-3000 optical system.

Light from the tungsten lamp is focused through an opening in the internal structure of the deuterium lamp. Light from the tungsten and deuterium lamps is then focused through the flow cell by the source lens. After exiting the cell, the light passes through the spectrograph lens to the filter paddle and is focused into the slit. The light then passes through the slit to the grating, where it is diffracted into the component wavelengths. Measurement of the light occurs at the photodiode array. Each diode measures a narrow portion of the spectrum.

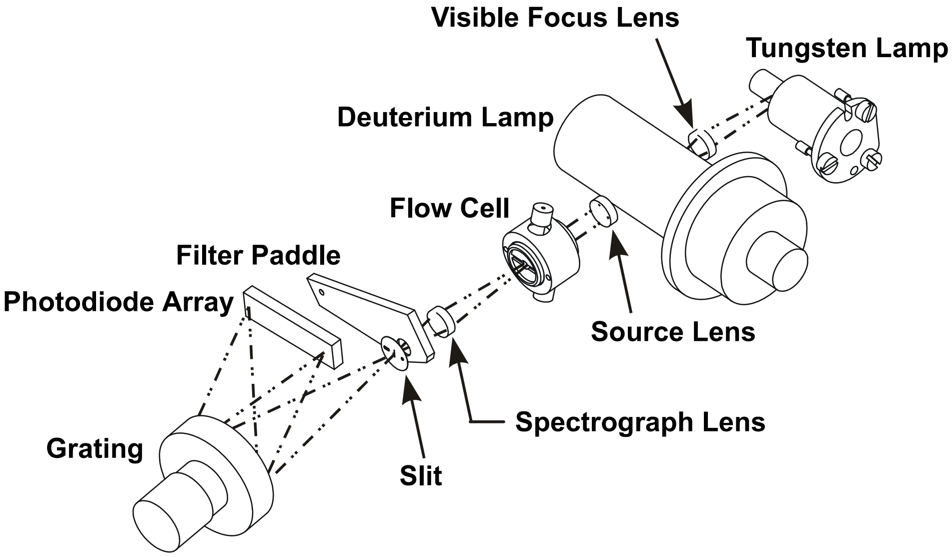


Fig. 5: PDA-3000 Optical System

Optical System Component	Function
Tungsten Lamp	The light source for visible and near-infrared wavelengths (380 to 800 nm). The tungsten lamp is focused through an opening in the internal structure of the deuterium lamp; this allows the lamps to share the optical axis to the source lens.
Visible Focus Lens	Focuses the visible light from the tungsten lamp to the through-hole of the deuterium lamp.
Deuterium Lamp	The light source for UV wavelengths (190 to 380 nm).
Source Lens	Receives the light from the tungsten and deuterium lamps and focuses it so that the beam passes through the flow cell.
Flow Cell	The measurement site for sample absorbance. The cell has a flat window on each side. Dionex offers five cells for use with the PDA-3000: analytical cells (in PEEK and 316 stainless steel), semi-micro cells (in PEEK and 316 stainless steel), and semipreparative cells (in PEEK only).

Optical System Component	Function
Spectrograph Lens	Receives the light from the flow cell and focuses it onto the slit.
Filter Paddle	The motorized filter paddle in the spectrograph has three positions: *Open* = Light passes, unobstructed, along the light path when the holmium oxide filter is moved out of the light path. *Blocked* = No light passes along the light path; used to measure the dark signal without turning off the lamps. *Holmium* = Places the holmium oxide filter in the light path; used to verify wavelength accuracy.
Slit	The width of the slit (equivalent to 1 nm) optimizes the optical resolution.
Grating	Diffracts the light beam into its component wavelengths and directs the light onto the photodiode array.
Photodiode Array	A series of 1024 photosensitive elements. Each element measures a narrow band of the spectrum. A spectrum is obtained by measuring the light intensity of each wavelength and reporting the results over the selected wavelength range.

2.4 Flow Cells

All flow cells are optimized for fast separations with no loss in chromatographic resolution. Note that the PDA is shipped without a flow cell. Flow cells must be ordered separately from Dionex. All cells are shipped with outlet fluidics, backpressure line and capillary to the column compartment and fittings.

For information about how to install, remove and clean flow cells, refer to section 6.2).

Important: Do not touch the cell windows. If you touch a window, clean it with isopropyl alcohol and a clean lens tissue.

Strong bases can etch the fused silica windows of the flow cell. If the mobile phase is a base, make sure the mobile phase concentration does not exceed 0.1 M. If the concentration of the base is greater than 50 mM, disconnect the separator column and flush the system with DI (deionized) water for 5 minutes at 1.0 mL/min immediately after the analysis. If strong base remains in the cell for 1 or 2 days, the cell windows may need to be replaced (→ section 6.2.5, page 89).

Do not use a PEEK flow cell with normal phase or chlorinated solvents; these solvents will damage the cell.

Do not use the stainless steel flow cell with low pH solutions; these solutions may cause corrosion, contamination, and metal leaching.

Analytical Flow Cell

Part No.	Cell Material	Cell Volume	Cell Path Length	Volume of Heat Exchanger + Inlet Tubing	Intended Flow Rate Range
6080.0200	PEEK	13 µL	10 mm	45 µL	0.5 to 5.0 mL/min
6080.0210	316 Stainless Steel	13 µL	10 mm	20 µL	0.5 to 5.0 mL/min

The analytical flow cell assembly (→ Fig. 6) is available in PEEK and 316 stainless steel. Two built-in heat exchangers—one on the cell inlet tubing and one on the cell handle—help stabilize the temperature of the mobile phase before it enters the cell.

The analytical cell is designed for applications with 3- to 4-mm ID separator columns and flow rates of 0.5 to 5.0 mL/min.

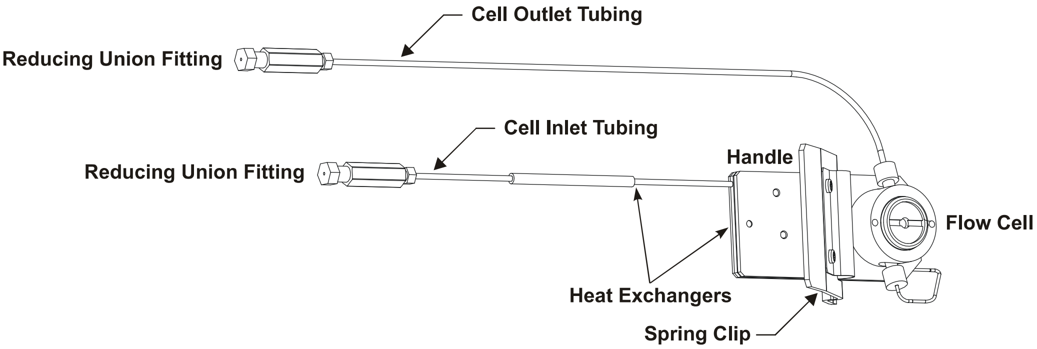

Cell Outlet Tubing
Reducing Union Fitting
Cell Inlet Tubing
Reducing Union Fitting
Handle
Flow Cell
Heat Exchangers
Spring Clip

Fig. 6: Analytical Flow Cell Assembly

Description	PEEK (6080.0200) P/N.	Stainless Steel (6080.0200) P/N
Reducing union fittings	6080.2101	6080.2102
Cell outlet tubing	6080.2401 0.38-mm (0.015-in) ID	6080.2403 0.25-mm (0.010-in) ID
Outlet fluidics kit, including capillary with tubing and fitting	6080.2410	

Semi-Micro Flow Cell

Part No.	Cell Material	Cell Volume	Cell Path Length	Volume of Heat Exchanger + Inlet Tubing	Intended Flow Rate Range
6080.0220	PEEK	3.1 µL	9 mm	5 µL	Up to 1.0 mL/min
6080.0230	316 Stainless Steel	3.1 µL	9 mm	5 µL	Up to 1.0 mL/min

The semi-micro flow cell assembly (→ Fig. 7) is available in PEEK and 316 stainless steel. Two built-in heat exchangers—one on the cell inlet tubing and one on the cell handle—help stabilize the temperature of the mobile phase before it enters the cell.

The semi-micro flow cell is designed for applications with 2- to 3-mm ID separator columns and flow rates of up to 1.0 mL/min.

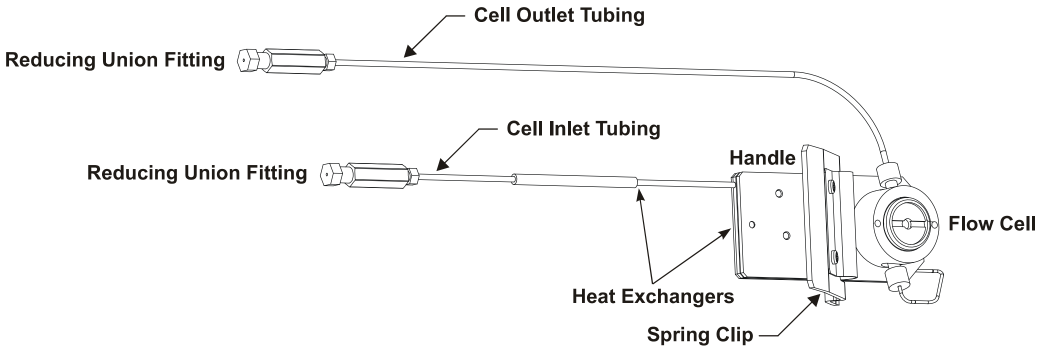

Cell Outlet Tubing
Reducing Union Fitting
Cell Inlet Tubing
Reducing Union Fitting
Handle
Flow Cell
Heat Exchangers
Spring Clip

Fig. 7: Semi-Micro Flow Cell Assembly

Description	PEEK P/N.	Stainless Steel Flow Cell P/N
Reducing union fittings	6080.2101	6080.2102
Cell outlet tubing	6080.2401 0.38-mm (0.015-in) ID	6080.2403 0.25-mm (0.010-in) ID
Outlet fluidics kit, including capillary with tubing and fitting	6080.2411	

Semipreparative Flow Cell

Part No.	Cell Material	Cell Volume	Cell Path Length	Volume of Heat Exchanger + Inlet Tubing	Intended Flow Rate Range
6080.0240	PEEK	0.7 µL	0.4 mm	N/A	5 to 100.0 mL/min

The semipreparative flow cell assembly (→ Fig. 8) is available in PEEK only. The cell is desinged for use with semipreparative flow applications and flow rates of 5.0 to 100.0 mL/min. The 0.4-mm path length minimizes detector absorbance for concentrated peaks.

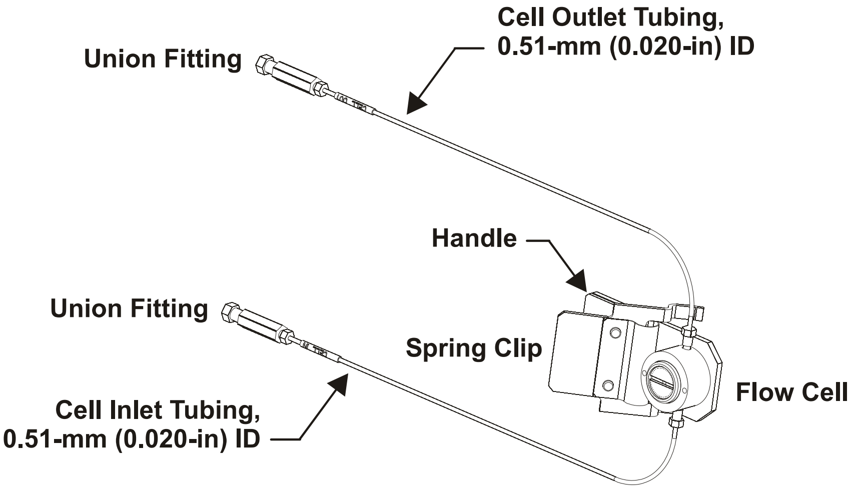

Cell Outlet Tubing, 0.51-mm (0.020-in) ID
Union Fitting
Handle
Union Fitting
Spring Clip
Flow Cell
Cell Inlet Tubing, 0.51-mm (0.020-in) ID

Fig. 8: Semipreparative Flow Cell Assembly

Description	PEEK P/N. (6080.0240)
Union fittings	6080.2103
Cell inlet or outlet tubing	6080.2406 0.51-mm (0.020-in) ID
Outlet fluidics kit, including capillary with tubing and fitting	6080.2412

2.5 Rear Panel

Fig. 9 illustrates the rear panel of the PDA-3000.

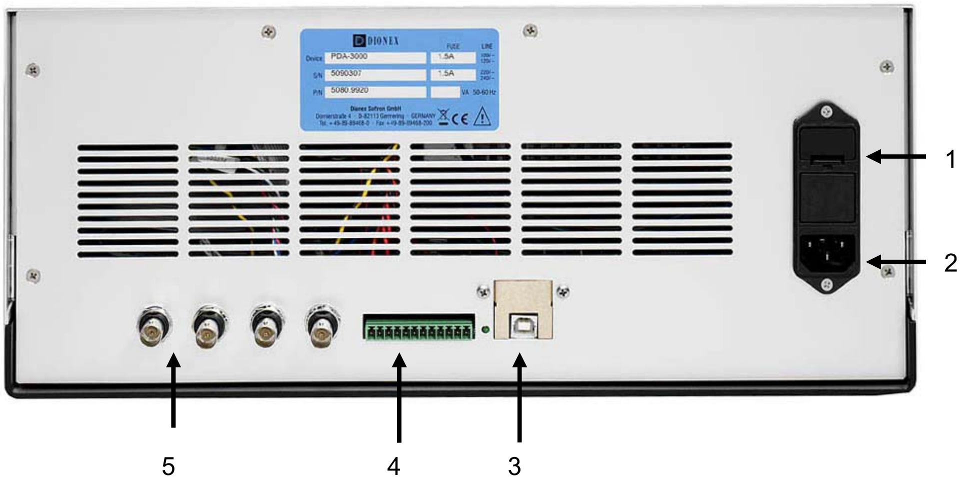


Fig. 9: PDA-3000 Rear Panel

No.	Description
1	Fuse holder The fuse holder holds the two main power fuses (→ section 6.4, page 97).
2	Power receptacle for connecting the power cord (→ section 3.5.4, page 28).
3	Analog outputs BNC connectors for connecting analog instruments (→ section 3.5.2, page 27).
4	Relay/TTL connector for controlling the detector during stand-alone operation (→ section 4.5, page 58).
5	USB port for connecting the detector to the Chromeleon PC (→ section 3.5.1, page 26).

2.6 Leak Sensor

Liquid leaks in the detector will be detected by the leak sensor that is installed on the inside front panel.

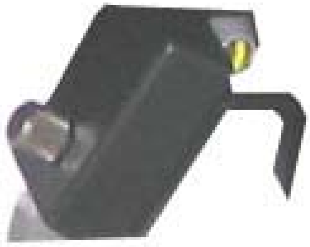


Fig. 10: Leak sensor

If the leak sensor detects a leak, the **Run** LED on the front panel door is red and an error message appears on the front panel display.

Eliminate the leak and dry the leak sensor as described in section 6.1 (→ page 83).

Tip: Inspect the detector for signs of leakage every day. Tighten or replace leaking connections if necessary.

2.7 Mobile Phases

Solvent quality significantly affects both detection limits and instrument performance. To ensure optimal performance of the PDA-3000, observe the following precautions.

Important: Strong bases can etch the silica windows of the flow cell. If the mobile phase is a base, make sure the mobile phase concentration does not exceed 0.1 M. If the concentration of the base is greater than 50 mM, disconnect the separator column and flush the system with DI (deionized) water for 5 minutes at 1.0 mL/min immediately after the analysis. If strong base remains in the cell for 1 or 2 days, the cell windows may need to be replaced (→ section 6.2.5, page 89).

Important: Do not use a PEEK flow cell with normal phase or chlorinated solvents; these solvents will damage the cell.

Important: Do not use the stainless steel flow cell with low pH solutions; these solutions may cause corrosion, contamination, and metal leaching.

- Prepare all mobile phases with spectroscopy-grade solvents, reagent-grade chemicals, and ASTM Type I (or better) filtered, DI (deionized) water.
- Degas all mobile phases before use and maintain them in a degassed state.
- Mobile phase pH affects not only the retention time of the separation, but the sample absorbance and the background absorbance of the mobile phase. If an analysis employs chemical suppression and compound detection techniques, compare the UV cutoff of the mobile phase before and after the suppressor to see whether it is advantageous to locate the PDA-3000 ahead of the suppressor. This location also limits suppressor exposure to backpressure.
- When changing from a buffer to a different operating mobile phase, be sure the solvents are miscible and will not induce precipitation of the buffers. Flush the cell with DI water immediately after the analysis.

2.8 Solvent Delivery System

- The pumping system should deliver continuous flow while maintaining a consistent mobile phase composition (if gradient elution is used). Fluctuations in system backpressure may cause baseline disruptions. High sensitivity applications require a smooth, low-pulsation pump. To ensure optimal results, follow the maintenance schedule recommended in the pump user's manual.
- All materials in the solvent delivery system must be fully compatible with the mobile phases required for the analysis. For example, IC and BioLC analyses frequently use mobile phases with extreme pH levels or high salt concentrations. A PEEK pumping system and PEEK flow cell are required when running these mobile phases because the presence of stainless steel in the flow path would contaminate the system.

- Reversed-phase solvents (methanol, moderate concentrations of acetonitrile, etc.) are acceptable for a PEEK system. Other solvents (and high concentrations of acetonitrile) require a stainless steel system.
- All tubing connections should be Teflon®, Tefzel®, PEEK, stainless steel, or titanium as required for the specific operating pressures and application.
- All mobile phase reservoirs should be compatible with the solvents used with your application. Some plasticizers in blow-molded components leach and are strong UV absorbers.
- Cleanliness is very important. After operating in detection modes that do not require optically clean reagents (such as conductivity), the solvent delivery system may need to be thoroughly cleaned. A basic cleaning procedure is described below; for additional information, refer to the pump user's manual.

Important: To avoid contamination, always wear gloves when handling pump components in the flow path. Contamination may cause baseline disruptions, spurious peaks, and inconsistent results, especially in sensitive applications.

To clean the pump and system between applications:

1. Remove the column and the suppressor (if installed).
2. Connect the line exiting the injection valve directly to the flow cell inlet tubing.
3. Flush the system with DI water at 1.0 mL/min for 20 minutes.
4. Flush the system with methanol at 1.0 mL/min for 20 minutes. For very sensitive applications, continue flushing for several hours.
5. Flush the system with DI water at 1.0 mL/min for 15 to 20 minutes.
6. Reinstall the column (and the suppressor, if removed) and equilibrate the system before resuming operation.

Important: If there is a heavy salt build-up on the pistons, remove the pistons and clean them with DI water. Refer to the pump user's manual for instructions. (Cleaning the pistons removes salt crystals that can abrade the piston, thereby causing the main seal to wear out prematurely and allow leaks.)

Important: Replace the piston seals if there is a heavy salt build-up on the pistons, if the seals leak, or if the pistons are replaced. Refer to the pump user's manual for instructions.

3 Installation

3.1 Facilities Required

- Make sure the PDA-3000 installation site meets the power and environmental specifications listed in section 7, page 97.
- Optical detectors are sensitive to vibrations. Install the PDA-3000 on a sturdy workbench in an environment that is vibration-free.
- Allow at least 6 cm (2.4 in) behind and to the sides of the detector for ventilation to cool the electronics and dissipate the heat generated by the lamps.
- Optical detectors are sensitive to changes in temperature and light. Protect the PDA-3000, the columns, and all tubing connections from drafts. Do not operate the PDA-3000 unless the front panel, lamp cover, and flow cell cover are in place.

3.2 Unpacking the PDA-3000

All electrical and mechanical components of the detector are carefully tested before the instrument is shipped from the factory. After unpacking, please inspect the instrument for any signs of mechanical damage, which might have occurred during transit.

NOTE: Immediately report any shipping damage to both, the incoming carrier and Dionex. Shipping insurance will compensate for the damage only if reported immediately.

Keep the original shipping container and packing material. They provide excellent protection for the instrument in case of future transit. Shipping the unit in any other packaging automatically voids the product warranty.

1. Place the shipping container on the floor and remove the accessories.
2. Grasp the detector by the sides. Slowly and carefully, pull the instrument out of the shipping container and place it on a stable surface.

Important: To prevent the detector from falling, always lift the unit by the sides. Do not lift the detector by the packing material or the front panel door.

Important: Afin d'empêcher l'instrument de tomber, saisissez-la par les côtés. Ne soulevez l'instrument à l'aide du matériau d'emballage ou par la porte du panneau avant.

3. Before connecting the detector to the power supply, wait approximately 1 hour to allow the instrument to come to room temperature and to allow any condensation that might have occurred during shipping to evaporate. After 4 hours, check the detector; if condensation still exists, allow the detector to continue to warm up (without connecting it to the power source) until the condensation is completely gone.

3.3 Positioning the Detector in the UltiMate 3000 System

If the detector is part of an UltiMate 3000 system, e.g., for analytical HPLC applications, Dionex recommends stacking the individual modules, for example, as shown in Fig. 11 (→ page 25) and interconnecting them on the rear panel. However, the arrangement of the system modules depends on the application.

NOTE: Apart from the solvent rack, all modules of the UltiMate 3000 system can be connected separately to the server PC. However, Dionex recommends interconnecting all modules, and then connecting the system to the Chromeleon server PC via only one connection. To do so, use the USB hub on the pump or detector.

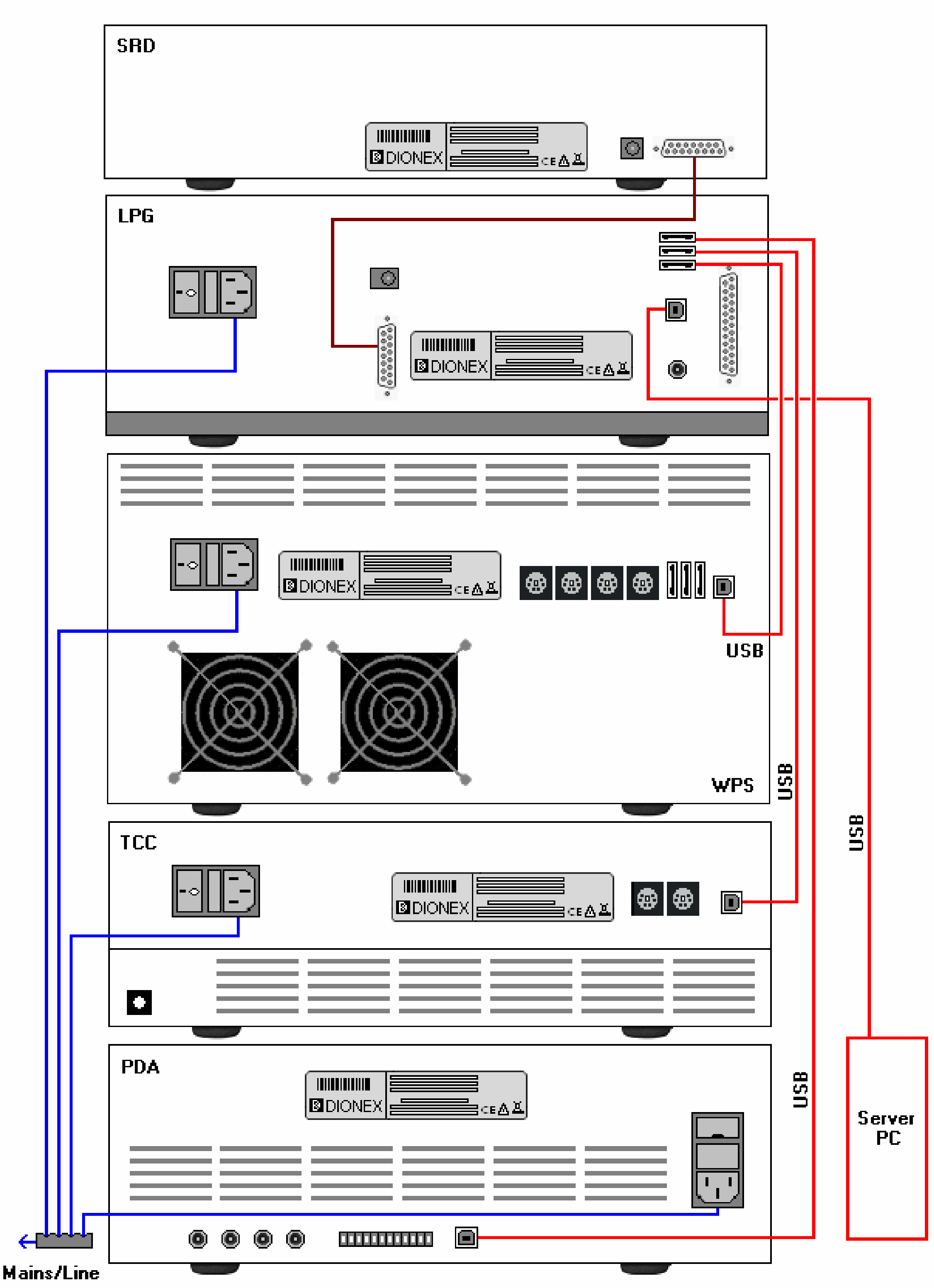

SRD
DIONEX
LPG
DIONEX
DIONEX
USB
WPS
USB
USB
TCC
DIONEX
PDA
DIONEX
USB
Server PC
Mains/Line

Fig. 11: Example for the rear panel connections on an UltiMate 3000 system

3.4 Installing Chromeleon Software

NOTE: Dionex strongly recommends installing Chromeleon *before* connecting the PDA-3000 to the computer. When you install the Chromeleon software first, the USB driver for the detector is automatically loaded and the Windows operating system can detect the detector when the power is turned on.

Before going on to 3.5, verify that Chromeleon is installed on the computer and a license is installed.

3.5 Connecting the Detector

3.5.1 USB Connection

The PDA-3000 can be connected directly to a USB port on the computer running Chromeleon.

NOTE: Apart from the solvent rack, all modules of the UltiMate 3000 system can be connected separately to the server PC. However, Dionex recommends interconnecting all modules, and then connecting the system to the Chromeleon server PC via only one connection. To do so, use the USB hub on the pump or autosampler.

1. Plug the "A" connector of a USB cable into the USB port on the computer (→ Fig. 12).
2. Plug the "B" connector of the USB cable into a USB receptacle on the PDA-3000 rear panel.

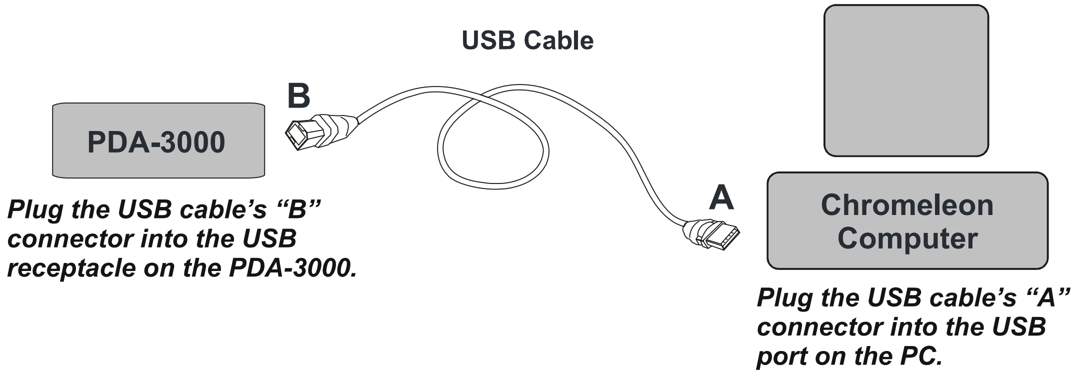

USB Cable
B
PDA-3000
A
Chromeleon Computer
Plug the USB cable's "B" connector into the USB receptacle on the PDA-3000.
Plug the USB cable's "A" connector into the USB port on the PC.

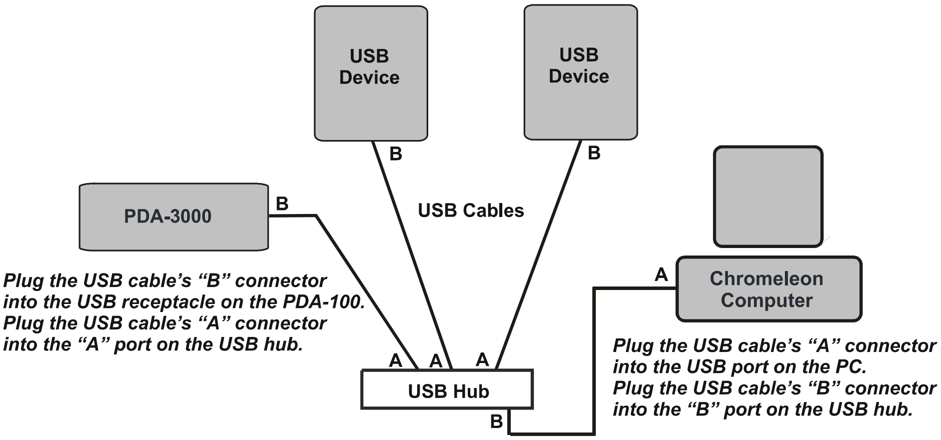
Fig. 12: PDA-3000 Connected Directly to the Chromeleon PC

If a USB port is not available on the PC, connect the PDA-3000 to an external USB hub and then connect the hub to the PC. The hub and extra USB cable required for this configuration are options and are not included with the PDA-3000.

Important: The USB standard limits the USB cable length to 5 meters (5.5 yds). Each USB device can be separated from the PC by no more than five hubs. Thus, if five hubs are installed, each USB device can be located no more than 30 meters (32 yds) from the PC.

Important: Carefully secure all USB cables, the USB hub, and the hub power cable so that they cannot be accidentally disconnected.

1. Plug the "A" connector of a USB cable provided in the PDA-3000 Ship Kit into a port on the external USB hub. Plug the "B" connector of the cable into the USB receptacle on the PDA-3000 rear panel (→ Fig. 13).
2. Plug the "A" connector of a standard USB cable into a USB port on the computer. Plug the "B" connector of the cable into the "B" port on the USB hub (→ Fig. 13).



Plug the USB cable's "B" connector into the USB receptacle on the PDA-100. Plug the USB cable's "A" connector into the "A" port on the USB hub.

Plug the USB cable's "A" connector into the USB port on the PC. Plug the USB cable's "B" connector into the "B" port on the USB hub.

Fig. 13: Three USB Devices Connected to a USB Hub

3.5.2 Analog Output Connection

Connect a recorder, an integrator, or an A/D data acquisition device to the **ANALOG OUT** connector on the PDA-3000 rear panel (→Fig. 9, page 19). The four PDA-3000 analog outputs are standard female BNC connectors and provide a 1 volt output maximum. The full-scale absorbance range can be set to 0.0005, 0.001, 0.01, 0.1, 0.5, 1.0, 2.0, or 3.0 AU full-scale in Chromeleon.

The outputs are controlled by absorbance wavelengths 1 through 4 in Chromeleon. The software controls the wavelength, sample bandwidth, reference wavelength, full-scale absorbance output, and offset within the 0 to 1 volt range.

The analog outputs continue to output data even after the computer running Chromeleon is turned off or is disconnected from the PDA-3000. When the detector power is turned on again, the analog output settings will be either the factory defaults (→ section 4.4, page 55) or the settings selected the last time the detector was controlled by the computer.

3.5.3 Relay/TTL Connections

The two TTL outputs and two Relay outputs on the instrument's rear panel (→Fig. 9, page 19) can be used in Chromeleon to exchange digital signals with external instruments. The accessories kit includes a 12-pin connector (part no. 6080.9010). To attach individual wires and twisted pairs to the connector, strip the end of the wire(s), insert into the connector, and tighten the locking screw(s) with the small screwdriver included in the ait.

The TTL and Relay outputs can be programmed as part of a PGM file (→ section 4.2.2, page 37) in Chromeleon, or controlled manually through a Control panel in the software.

For information about the port and pin assignment, refer to the technical appendix (→ page 103).

⚠ **Caution**: Connection of relay loads and their power sources to the TTL outputs will damage the TTL output stage. If the relay load can source more than 200 mA at 5V or higher, it may damage the Data Processing board.

⚠ **Warning**: SHOCK AND FIRE HAZARD—Inputs and outputs are not rated for connection to an AC power main. DO NOT CONNECT TO 100, 115, 220, 230, OR 240 VAC POWER SOURCES; FIRE OR ELECTROCUTION MAY RESULT.

⚠ **Avertissement**: DANGER D'ÉLECTROCUTION ET D'INCENDIE—Le module d'alimentation électrique fonctionne aux potentiels du secteur. Faites effectuer toutes les réparations par un personnel qualifié.

3.5.4 Connecting the Power Cord

The PDA-3000 power entry is socketed for a modular power cord (IEC 320 C13). The detector operates from input voltages over a range of 85 to 265 VAC, 47 to 63 Hz power, and requires a grounded, single-phase power source. The typical input power is 100 W and the maximum line draw is 3.5 amps at 110 VAC (when the detector power is turned on). The appropriate line voltage and frequency are selected automatically.

⚠ **Warning**: SHOCK HAZARD—To avoid electrical shock, use a grounded receptacle. Do not operate the PDA-3000 or connect it to AC power mains without an earthed ground connection.

⚠ **Caution**: The power supply cord is used as the main disconnect device. Make sure the socket-outlet is located near the PDA-3000 and is easily accessible.

⚠ **Avertissement**: DANGER D'ÉLECTROCUTION—Pour éviter toute électrocution, il faut utiliser une prise de courant avec prise de terre. Ne l'utilisez pas et ne le branchez pas au secteur C.A. sans utiliser de branchement mis à la terre.

⚠ **Mise en garde**: Le cordon d'alimentation principal est utilisé comme dispositif principal de débranchement. Veillez à ce que la prise de base soit située/installée prês du module et facilement accessible.

Connect a modular power cord (IEC 320 C13) from the PDA-3000 main power receptacle (→Fig. 9, page 19) to a grounded, single-phase power source. Use the front panel **Power** switch to turn the detector on and off.

3.6 Setting Up the Detector in Chromeleon

This section provides brief instructions for setting up Chromeleon. For details about any of these steps, refer to the Chromeleon Help.

NOTE: Dionex recommends installing the Chromeleon software before connecting the detector to the USB port on the Chromeleon computer. When you install the Chromeleon software first, the Windows operating system will detect the detector automatically when the detector power is turned on.

3.6.1 Loading the USB Driver for the Detector

1. Turn on the computer power, if it is not already on.
2. If this is a local computer, log on to the Windows operating system (XP or 2000) as a local administrator. If this is a network computer, log on as a user with local computer administrator privileges.
3. Start the Chromeleon **Server Monitor** program by double-clicking the Chromeleon Server Monitor icon on the Windows taskbar.

   If the Server Monitor icon is not on the taskbar, click **Start** on the taskbar, point to **Programs** (or **All Programs**, depending on the operating system), point to **Chromeleon,** and then click **Server Monitor**.
4. Click **Start** to start the server.
5. Click **Close** to close the Server Monitor window. The Server Monitor icon appears on the taskbar.

   NOTE: Clicking the **Quit Monitor** button quits (exits) the Server Monitor program, but does not stop the server. To stop the server, click **Stop**.
6. Turn on the power of the detector.

7. Microsoft Windows will automatically detect the new detector and launch the **Found New Hardware Wizard**, which guides you through the USB installation. Select the following options:

   a) Accept the default option (Install the software automatically) and click **Next>**.

   b) Click **Finish** when the wizard reports that the software for the detector has been installed.

   If Windows fails to detect the detector and a message box asks for a USB configuration file (cmwdmusb.inf), this indicates that you connected the detector to the computer and turned on the power for the first time **before** you installed Chromeleon.

   To resolve the problem:

   a) Click Cancel in the Windows message box.

   b) Turn off the detector and unplug the USB cable from the computer.

   c) Install Chromeleon.

   d) Reconnect the USB cable to the computer and turn on the power to the detector. Windows will now automatically detect the detector and launch the **Found New Hardware Wizard**.

3.6.2 Installing the Detector

After the USB software for the detector has been installed (→ page 29), install and configure the detector in Chromeleon:

1. Start the Chromeleon **Server Monitor** if it is not already running (→ page 29).
2. Start the Chromeleon **Server Configuration** program by clicking **Start** on the taskbar. Point to **Programs** (or **All Programs**, depending on the operating system), point to **Chromeleon**, and then click **Server Configuration**.
3. If necessary, click the plus sign beside the server name to display the items underneath.
4. Select the timebase to which the autosampler will be assigned, or create a new timebase (on the **Edit** menu, click **Add Timebase**).
5. Open the **Add device to timebase** dialog box. To do so, click **Add Device** on the **Edit** menu or right-click the timebase and click **Add Device** on the menu.
6. On the **Manufacturers** list, click **Dionex HPLC: UltiMate 3000** and on the **Devices** list, click **PDA-3000 Detector**.
7. Chromeleon connects to the detector and transfers the settings from the instrument firmware to Chromeleon, setting the options installation tab pages accordingly.

8. On each tab page, verify that the settings are correct and select additional settings if needed. For a description of the tab pages click the **Help** button.
9. Click **Finish** to complete the installation of the autosampler.
10. On the **File** menu, click **Save Installation** and then close the Server Configuration program.

3.7 Installing the Flow Cell

The detector is shipped without a flow cell. Before initial operation of the detector, install a flow cell that is appropriate for the respective application. Flow cells must be ordered separately from Dionex.

Important: Do not touch the cell windows. If you touch a window, clean it with isopropyl alcohol and a clean lens tissue.

NOTE: No tools are required for removing and installing the flow cell. The knurled head screws on the flow cell cover and tubing bracket can be loosened and tightened by hand.

1. Open the front panel door.
2. Remove the two knurled head screws on the flow cell cover, and then remove the cover. (→Fig. 4, page 13).
3. **Analytical or semi-micro cell:** Remove the two knurled head screws on the tubing bracket to remove the bracket from the lamp cover (→Fig. 30, page 88).
4. Squeeze the handle of the flow cell and insert it straight into the optical bench. Release the handle, and then pull it forward slightly. If there is a click, the cell is properly mounted. If there is no click, rotate the handle slightly just until the cell clicks into place.

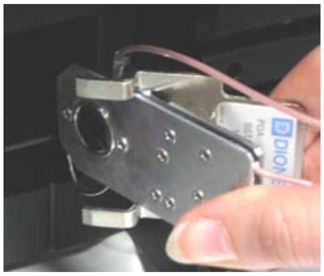


Fig. 14: Inserting the flow cell into the optical bench

5. Thread the flow cell inlet tubing through the tubing guide on the lamp cover and reinstall the tubing bracket (→Fig. 30, page 88).

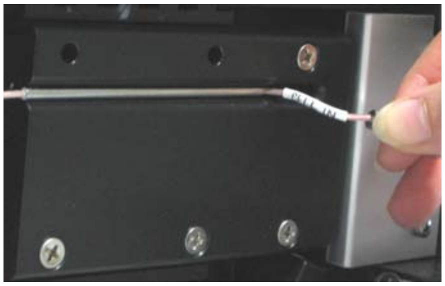


Fig. 15: Threading the inlet tubing through the tubing guide

6. Thread the flow cell outlet tubing through the slot on the side of the flow cell cover. Replace the cover and tighten the two screws on the cover fingertight.

Important: Do not pinch the tubing under the flow cell cover.

7. *(Optional)* Connect a backpressure line to the cell outlet connection. The backpressure line will provide enough restriction to generate the small amount of backpressure (0.14 to 0.34 MPa; 20 to 50 psi) required to help prevent bubbles from forming in the cell. If you decide to connect a backpressure line, you do not have to connect an outlet capillary as described in step 8.

   a) The flow cell is shipped with a 2-meter 0.25-mm (0.010-in) ID capillary for the backpressure line.

   b) Cut the tubing to the required length. The backpressure line should be 1 meter long for a flow rate of 1.0 mL/min, 2 meters for a flow rate of 0.5 mL/min, and so on.

   c) Connect the backpressure line to the cell out connection.

Important: The backpressure on the cell must not exceed 21 bar (300 psi) for a PEEK analytical or semi-micro cell, 35 bar (500 psi) for a stainless steel analytical or semi-micro cell, or 100 bar(1450 psi) for a semipreparative cell.

8. The flow cell is shipped together with a 0.5 m capillary and hose to be connected to the cell outlet connection. Connect this liquid line to the cell out connection.

Important: Make sure not to twist the end of the flow cell inlet and outlet tubings when connecting them from /to the liquid line connections. Preferably use a tool to hold the union fitting while you remove or install any connections.

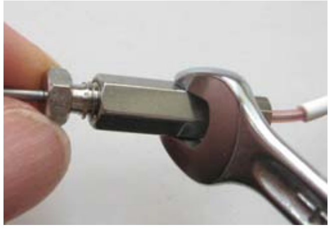


Fig. 16: Union fitting

9. The detector enclosure provides four slots (→ Fig. 4, page 13). Thread the lines connected to the flow cell in and out connections through these slots to prevent them from being pinched when the front panel door is closed.
10. Close the front panel door.
11. Connect the cell inlet connection to the separator column outlet.

3.8 Connecting the Waste Tubing

Connect the waste tubing (part no. 6000.5001) to the outlet at the bottom right (see figure below). Place the free end of the waste tubing in a waste container below the level of the workbench on which the PDA-3000 is installed.

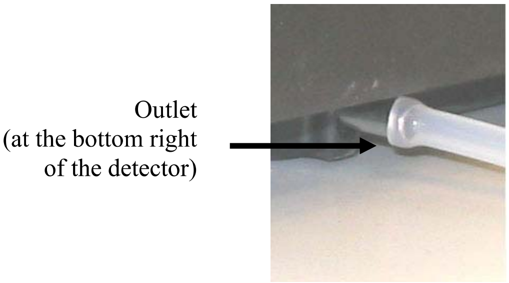


Abb. 17: Outlet

Important: The tube must remain below the drain port. If the tube is above the drain port, the drip tray may overflow inside the detector.

If the detector is part of an UltiMate 3000 system, you may connect the drain tubing of the system modules with the connecting pieces from the devices' accessories kits:

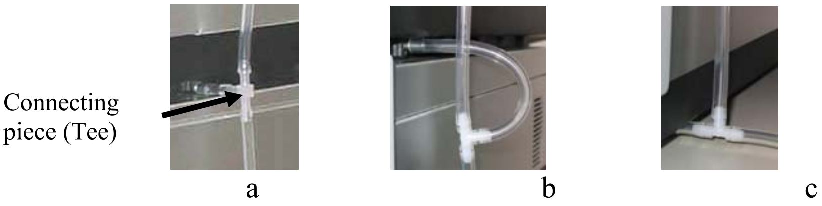


Fig. 18: Connected drain tubing

Connect the drain tubing from the solvent rack to the pump and from the pump to the detector as shown in Fig. 18 a. Dionex recommends connecting the drain tubing from the column compartment as shown in Fig. 18 b. This is to avoid that the gas sensor in the column compartment is influenced by the drain tubing from the other modules. As an alternative, you may direct the drain tubing from the Tee of the column compartment separately to the waste container. Connect the drain tubing from the detector as shown in Fig. 18 c.

Dionex recommends cutting the tubing, which is provided in the instrument's accessories kit, to the following lengths:

Tubing from	**Length (mm)**
Solvent rack to pump	215
Pump to detector	332
Pump or detector to Tee	30
Detector to the column compartment	217
Column compartment to Tee	133
Column compartment (Tee) to detector	110

4 Detector Control

The PDA-3000 Photodiode Array Detector is designed for use with IC (ion chromatography), BioLC® (biocompatible liquid chromatography), and HPLC (high-performance liquid chromatography) systems and should not be used for any other purpose. Operation of the PDA-3000 in a manner not specified by Dionex may result in personal injury. If you have a question regarding appropriate usage, contact Dionex Service before proceeding.

4.1 Getting Started

Before beginning operation of the PDA-3000, follow these steps:

1. Press the Power switch on the front panel to turn on the detector (→Fig. 3, page 11).
2. Verify that the Power LED is lighted.
3. Verify that the Deuterium and/or Tungsten LEDs are lighted.
4. Wait 20 to 30 minutes for the lamps to stabilize. (For sensitive applications or for detector validation, allow at least 2 hours. After installing a new lamp, allow 8 to 24 hours.)

4.2 Routine Operation by Chromeleon

Two modes of software control are available:

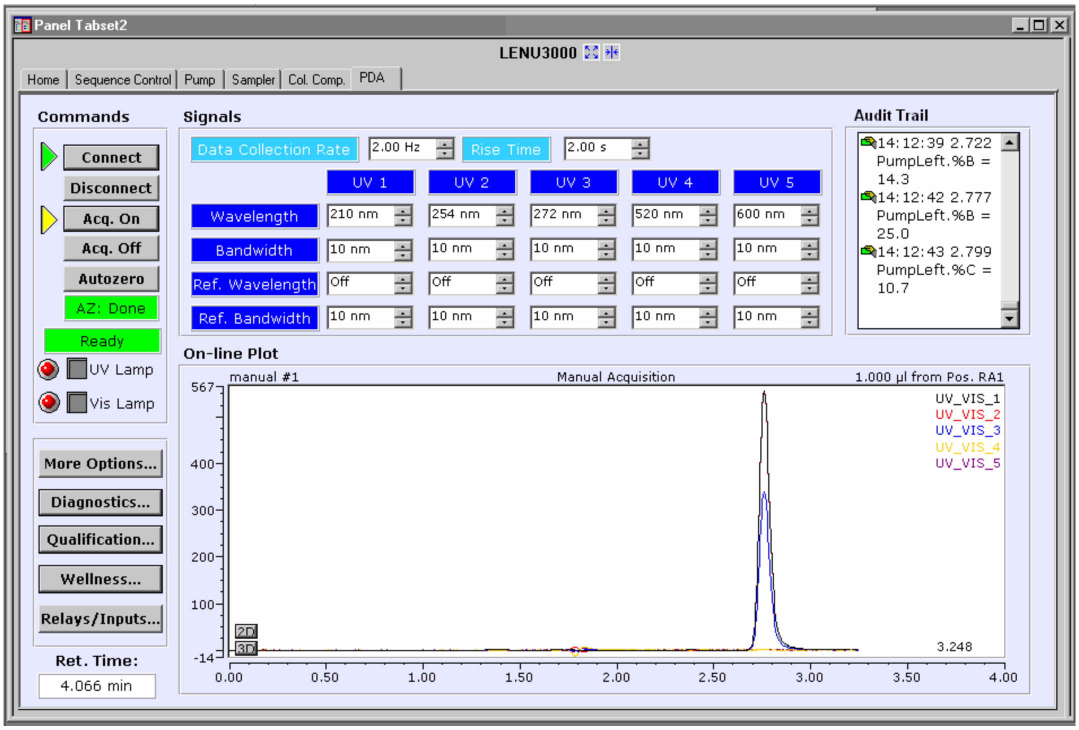
- **Direct Control** (→ section 4.2.1)
  With direct control, you select operating parameters and commands from control panels or the **Commands** (F8) dialog box. Direct commands are executed as soon as they are entered. (To open the **Commands** dialog box, open a Chromeleon control panel. Select **Command** on the **Control** menu. The **Commands** dialog box is opened. You can also open this box by pressing the F8 key. To display the commands and properties that are available for the detector, click the "+" sign beside **UV** in the left list box.)
- **Automated Control** (→ section 4.2.2)
  With automated control, you create a program (or PGM File). This is a list of control commands, executed in chronological order, for automated operation of the detector. Programs can be created automatically with the help of a software wizard or manually by editing an existing program.

For details about how to control the detector, refer to the *Chromeleon online Help* and/or *User Manual*.

4.2.1 Direct Control

In the direct control mode, PDA-3000 commands and operating parameters are selected from the Chromeleon menu bar, toolbars, and Control panels. Direct control commands are executed as soon as they are entered. Parameters can be modified at any time during manual data acquisition.

1. Start the Chromeleon Server by double-clicking the Server Monitor icon on the taskbar.
2. Start Chromeleon by selecting **Start** > **Programs** > **Chromeleon** > **Chromeleon**. The main window and the Browser will appear.
3. Select **Default Panel Tabset** from the **View** menu or click the icon on the standard toolbar. The **Connect to Chromeleon Server** dialog box opens.
4. Select the chromatography server on which the chromatography system (timebase) you want to control is configured and click **OK**. The panel tabset opens. The panel tabset consists of a set of default control panels, organized on tab pages. If there are multiple chromatography systems connected to the selected server, the window displays a panel tabset for each system.
5. Click the PDA tab to open the PDA-3000 control panel.


Panel Tabset2
LENU3000
Home
Sequence Control
Pump
Sampler
Col. Comp.
PDA
Commands
Connect
Disconnect
Acq. On
Acq. Off
Autozero
AZ: Done
Ready
UV Lamp
Vis Lamp
More Options...
Diagnostics...
Qualification...
Wellness...
Relays/Inputs...
Signals
Audit Trail
On-line Plot
manual #1
Manual Acquisition
1.000 µl from Pos. RA1
UV_VIS_1
UV_VIS_2
UV_VIS_3
UV_VIS_4
UV_VIS_5
2D
3D
3.248
567
400
300
200
100
-14
0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00

Data Collection Rate: 2.00 Hz
Rise Time: 2.00 s

	UV 1	UV 2	UV 3	UV 4	UV 5
Wavelength	210 nm	254 nm	272 nm	520 nm	600 nm
Bandwidth	10 nm	10 nm	10 nm	10 nm	10 nm
Ref. Wavelength	Off	Off	Off	Off	Off
Ref. Bandwidth	10 nm	10 nm	10 nm	10 nm	10 nm

```
14:12:39 2.722
PumpLeft.%B =
14.3
14:12:42 2.777
PumpLeft.%B =
25.0
14:12:43 2.799
PumpLeft.%C =
10.7
```

Ret. Time: 4.066 min

Fig. 19: PDA-3000 Control Panel Example

6. Select the operating parameters (→ section 4.3, page 44).
7. Go to the Pump tab and turn on the pump. Set the flow rate and mobile phase composition.
8. Click **Acq. On** to begin manual data acquisition.
9. Click **Acq. Off** to stop manual data acquisition after the desired run time.
10. Data collected during the run may be saved to a sequence. To retrieve the data, select the assigned sample name within the sequence.

4.2.2 Automated Control

A PGM file is a list of time-precise control commands, created by the user, for automated operation of the PDA-3000. PGM files can be created automatically (with the help of the PGM Wizard) or manually (by editing an existing PGM file).

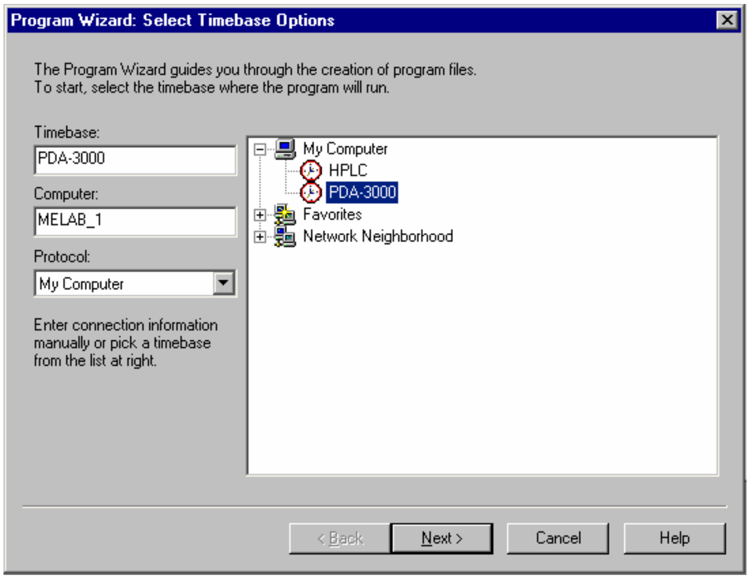
1. Start the Chromeleon Server by double-clicking the Server Monitor icon on the taskbar.
2. Start Chromeleon by selecting **Start > Programs > Chromeleon > Chromeleon**. The main window and the Browser will appear.

Creating a New PGM File

Careful selection of the data collection rate, rise time, and other operating parameters will optimize the performance of the PDA-3000. Before creating a PGM file, you may want to review section 4.2.3 (→ page 42) for a description of these parameters, how they interact with each other, and some guidelines for their selection.

1. Select **File > New**.
   A dialog box appears.
2. Select **Program File** and click **OK**.

   The initial Program Wizard screen is displayed.


Program Wizard: Select Timebase Options

The Program Wizard guides you through the creation of program files.
To start, select the timebase where the program will run.

Timebase: PDA-3000

Computer: MELAB_1

Protocol: My Computer

Enter connection information manually or pick a timebase from the list at right.

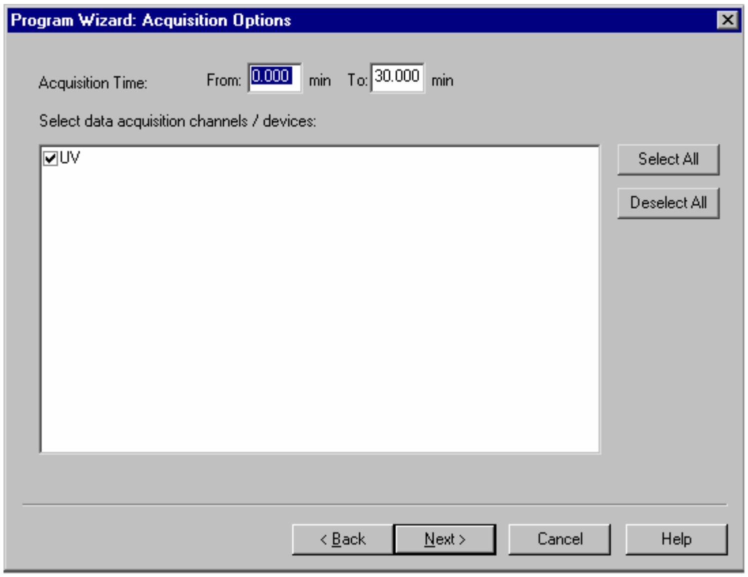
My Computer
HPLC
PDA-3000
Favorites
Network Neighborhood
< Back
Next >
Cancel
Help

Fig. 20: Program Wizard: Selecting the Timebase

3. Select the **Timebase** where the program will run.
4. Click **Next** to continue.

   If the selected timebase includes other devices (pump, detector, etc.), Program Wizard screens for these devices precede the PDA-3000 screens.

   Click the **Next** button on each screen for another device until the **Acquisition Options** screen appears.


Program Wizard: Acquisition Options

Acquisition Time: From: 0.000 min To: 30.000 min

Select data acquisition channels / devices:

☑ UV

Select All
Deselect All
< Back
Next >
Cancel
Help

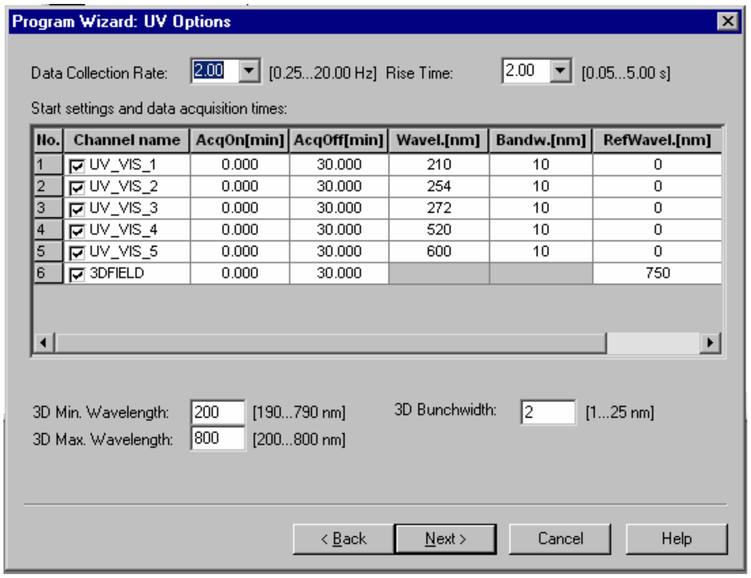
Fig. 21: Program Wizard: Acquisition Options

5. To determine for how long data will be collected, specify when the **Acquisition Time** starts and ends.

6. Select the channels and/or devices for which data will be collected. To enable or disable all entries listed here, click **Select All** or **Deselect All**, respectively.

7. Click **Next** to continue.

The following screen is displayed.



Data Collection Rate: 2.00 [0.25...20.00 Hz] Rise Time: 2.00 [0.05...5.00 s]

Start settings and data acquisition times:

No.	Channel name	AcqOn[min]	AcqOff[min]	Wavel.[nm]	Bandw.[nm]	RefWavel.[nm]
1	☑ UV_VIS_1	0.000	30.000	210	10	0
2	☑ UV_VIS_2	0.000	30.000	254	10	0
3	☑ UV_VIS_3	0.000	30.000	272	10	0
4	☑ UV_VIS_4	0.000	30.000	520	10	0
5	☑ UV_VIS_5	0.000	30.000	600	10	0
6	☑ 3DFIELD	0.000	30.000			750

3D Min. Wavelength: 200 [190...790 nm] 3D Bunchwidth: 2 [1...25 nm]

3D Max. Wavelength: 800 [200...800 nm]

Fig. 22: Program Wizard: UV Options

8. Enter the **Data Collection Rate** and the **Rise Time**.

NOTE: When the data collection rate is set to 10.0 Hz, the 0.05 second rise time setting is not available. When the data collection rate is set to 20.0 Hz, the 5.0 second rise time setting is not available.

NOTE: The 20.0 Hz data collection rate and 0.05 second rise time settings are not supported unless PDA-3000 Firmware version 1.1.0 (or later) is installed.

9. To program **UV_VIS_1** through **UV_VIS_5** (the single-wavelength channels):

   a) Check (or clear) a check box to determine whether 2D data from this channel will be collected.

   b) For each selected channel, enter the starting and ending data acquisition times as well as the wavelength, bandwidth, reference wavelength, and reference bandwidth.

NOTE: The settings for these channels apply to both analog outputs and ditigal outputs. UV channel 1 = analog output, UV2 = analog output 2, etc.

NOTE: When the reference wavelength is off, the reference bandwidth is not used. The reference value is the first data point of the sample wavelength and bandwidth after autozero.

10. To program **3DFIELD** (the 3D channel):

    a) Check (or clear) a check box to determine whether 3D data will be collected.

    b) If the channel is selected, enter the starting and ending data acquisition times as well as the reference wavelength and reference bandwidth.

NOTE: The reference wavelength (if set) must be within the 3D range. The total of the reference wavelength plus one-half the reference bandwidth must always be within the sample wavelength range selected; if it is not, the reference wavelength will default to the longest wavelength.

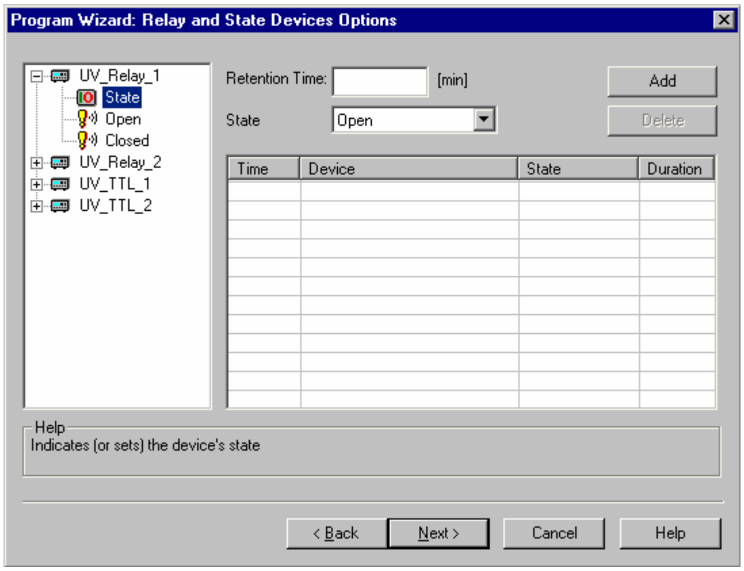
11. Enter the **3D Min. Wavelength** and **3D Max. Wavelength**.

NOTE: If the data collection rate is 20 Hz, the 3D sample wavelength range must be ≤ 310 nm.

12. Enter the **3D Bunchwidth** (the distance between wavelengths).

13. Click **Next** to continue.

The following screen is displayed. The tree control lists states for all devices in the selected timebase. Click the plus sign to the left of a device name to expand or collapse the struture and view the device states.


Program Wizard: Relay and State Devices Options
UV_Relay_1
State
Open
Closed
UV_Relay_2
UV_TTL_1
UV_TTL_2
Retention Time:
[min]
Add
Delete
Time
Device
State
Duration
< Back
Next >
Cancel
Help

State: Open

Help
Indicates (or sets) the device's state

Fig. 23: Program Wizard: Relay and State Device Options

NOTE: If you have already selected these parameters on the device page for a particular module, the list box on the Relay and State Devices Options page already contains this information. If you change any settings here, Chromeleon will automatically copy the changes to the appropriate device page.

14. Select a device in the tree control.

15. (Optional) Enter the **Retention Time** for the selected device. If you do not enter a time, the entry appears in the initialization section of the PGM file.

16. Specify the **State** or **Duration**:
    - Clicking **State** in the tree control specifies a simple switch command. Select **Open** or **Closed** from the drop-down list box.
    - Clicking a state description in the tree control sets the device to the selected state and lets you specify a **Duration**. Enter the duration of the selected state in the edit box.

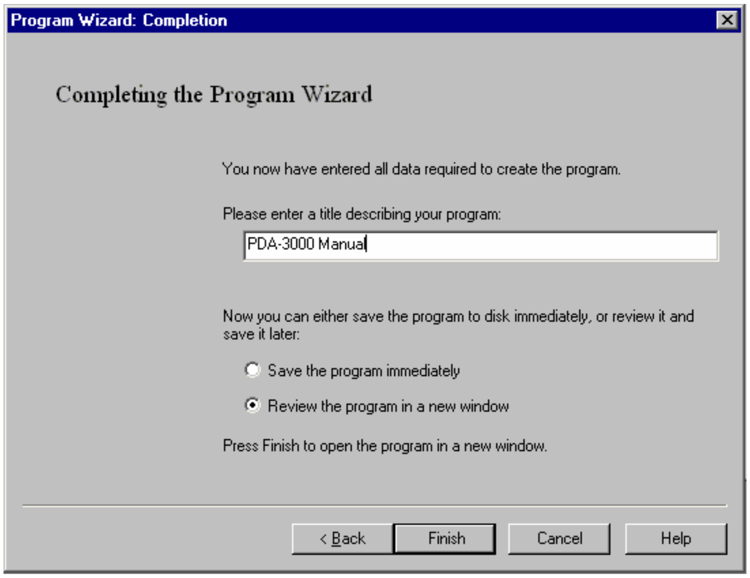
17. To add the following information to the list box, click **Add**:
    - **Retention Time** value
    - Name of the selected **Device**
    - **State** or **Duration** value

    Chromeleon will automatically organize all entries in the list box in chronological order.

    To remove an entry from the list box, select the entry and click **Delete**. (All entries in the list box will automatically be reorganized.)

18. Click **Next** to continue.

    The following screen is displayed.



Program Wizard: Completion

Completing the Program Wizard

You now have entered all data required to create the program.

Please enter a title describing your program:

PDA-3000 Manual

Now you can either save the program to disk immediately, or review it and save it later:

○ Save the program immediately

● Review the program in a new window

Press Finish to open the program in a new window.

Fig. 24: Program Wizard: Finishing the program

19. (Optional) Type a descriptive **Title** for the program.

20. Select one of the following options

- To save the programm without first reviewing it, select **Save the program immediately**. Clicking **Finish** opens a dialog box in which you can save the program.
- To review the program before saving it, select **Review the program in a new window**. Clicking **Finish** opens the program in the PGM Editor. Use the PGM Editor to review the program and to manually edit it, if necessary. When you finish, exit the PGM Editor. This displays a prompt asking whether the program should be saved; clicking **Yes** opens a dialog box in which you can save the program.

4.2.3 Control Commands

Chromeleon includes many control commands that are available for all detectors. In addition, there are commands that are exclusively for the PDA-3000 (see the table below). These commands can be selected from either the default Control panel or the Commands dialog box. (To open the Commands dialog box, press **F8** or select **Command** on the **Control** menu.)

NOTE: For information about control commands, also refer to the Chromeleon Online Help.

PDA-3000 Command	Function
Average	When Average is On, Chromeleon averages the data points between Step intervals (→ page 53).
Bandwidth	Sets the bandwidth (the wavelength range at which the chromatogram is measured) of the sample wavelength (→ page 48).
BunchWidth	Determines how many nanometers are averaged when collecting 3D data. Bunching (or averaging) the signals of adjacent wavelengths reduces the size of the 3D data stored (→ page 52).
Data_Collection_Rate	Sets the rate at which Chromeleon collects digital data points from the detector (→ page 46).
NegativeAbsorptionLevel	Adjusts the negative absorbance; this permits the baseline and peaks to be seen even when the measured absorbance becomes negative after the Autozero (→ page 54).
Offset_Level	Sets the offset applied to the recorder output.
RefBandwidth	Sets the bandwidth of the reference wavelength, if one is selected. If RefWavelength is **Off**, the RefBandwidth setting has no effect (→ page 50).
RefWavelength	Sets the reference wavelength. If RefWavelength is **Off**, the reference value is the first data point of the sample wavelength and bandwidth after autozero is performed (→ page 48).
Rise_Time	Sets how quickly the detector responds to a change in signal (→ page 45).

PDA-3000 Command	Function
Step	Sets the step, which is the time interval between two successively stored data points in the chromatogram (→ page 52).
TTL_Input_Mode	Sets the TTL input signal mode.
UV_Lamp	Turns the deuterium lamp on and off.
UVLampAge	Reports (or resets) the number of hours the deuterium lamp has been on.
UVLampPowerOnSetting	Selects the power-up setting for the deuterium lamp (i.e., whether the lamp is turned on whenever the detector is turned on). The default setting is On.
Visible_Lamp	Turns the tungsten lamp on and off.
VisLampAge	Reports (or resets) the number of hours the tungsten lamp has been on.
VisLampPowerOnSetting	Selects the power-up setting for the tungsten lamp (i.e., whether the lamp is turned on whenever the detector is turned on). The default setting is On.
Wavelength	Sets the sample wavelength (up to five sample wavelengths can be programmed) (→ page 47).
WavelengthCal	Runs the wavelength calibration procedure to improve wavelength accuracy in the UV range.
WavelengthCalResult	The result of the last wavelength calibration procedure.
WavelengthVerTest	Performs the wavelength verification test via the holmium oxide filter.
WavelengthVerTestResult	The result of the last wavelength verification test.

4.3 Optimizing Detector Performance

The performance of the PDA-3000 can be optimized by careful selection of key operating parameters. This section defines these parameters, describes how they interact with each other, offers guidelines for their selection, and includes example chromatograms.

The following table summarizes the topics discussed in this section.

Operating Parameter	**Performance Characteristics Affected**
Flow cell material	Chemical compatibility
Rise time	Peak resolution, sensitivity, baseline noise
Data collection rate	Peak resolution, disk space
Sample wavelength	Sensitivity, linearity
Bandwidth	Sensitivity vs. baseline noise
Reference wavelength	Baseline drift
Reference bandwidth	Baseline noise, baseline drift
Bunch width	Spectral resolution, peak match, disk space
Step*	Baseline noise, rise time, data collection rate, disk space
Average*	Rise time, data collection rate
Negative absorbance	Peak detection, baseline noise, linearity
* Set automatically by Chromeleon.	

4.3.1 Flow Cell Material

The flow cell materials must be chemically compatible with the mobile phases and analytes of interest.

Important: Strong bases can etch the fused silica windows of the flow cell. If the mobile phase is a base, make sure the mobile phase concentration does not exceed 0.1 M. If the concentration of the base is greater than 50 mM, disconnect the separator column and flush the system with DI (deionized) water for 5 minutes at 1.0 mL/min immediately after the analysis. If strong base remains in the cell for 1 or 2 days, the cell windows may need to be replaced (→ section 6.2.5, page 89).

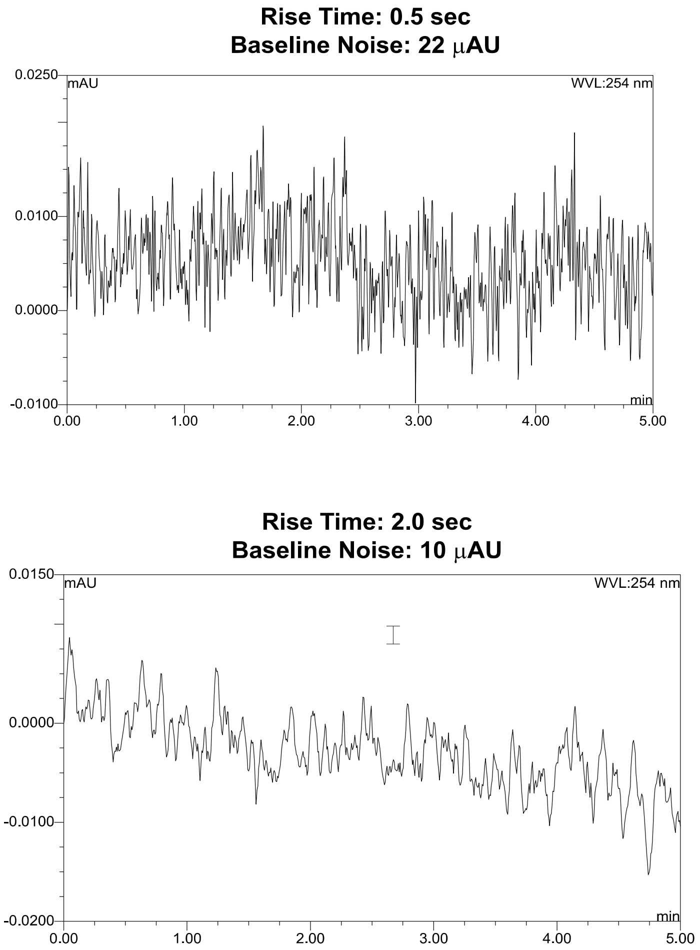
Important: Do not use a PEEK flow cell with normal phase or chlorinated solvents; these solvents will damage the cell.

Important: Do not use the stainless steel flow cell with low pH solutions; these solutions may cause corrosion, contamination, and metal leaching.

4.3.2 Rise Time

Rise time is a measure of how quickly the PDA-3000 responds to a change in signal. The rise time settings are 0.05, 0.1, 0.2, 0.5, 1.0, 2.0 (default), or 5.0 seconds. The selected rise time is applied to all data collected, and to both analog and digital output.

Select a rise time that is approximately 25% of the peak width at half-height of the narrowest peak of interest. A longer rise time allows more averaging of the signal and results in less short-term noise (→ Fig. 25, page 46). However, a rise time that is too long may result in reduced peak heights and asymmetric peak shapes. When set correctly, the rise time significantly reduces baseline noise, but reduces peak height only slightly.


Rise Time: 0.5 sec
Baseline Noise: 22 µAU

Rise Time: 2.0 sec
Baseline Noise: 10 µAU

Fig. 25: Effect of Rise Time on Baseline Noise

4.3.3 Data Collection Rate

The data collection rate (or *sample rate*) is the number of data points per second (Hz) at which the computer stores data from the PDA-3000. (The detector electronics always collect data at up to 20.0 Hz.)

The data collection rate settings are 0.25, 0.5, 1.0, 2.0, 2.5 (default), 5.0, 10.0 Hz or 20.0 Hz. The maximum number of data points is stored at 20.0 Hz. Slower data collection rates store the average of the 10.0 Hz points; for example, a 5 Hz rate stores the average of four 20.0 Hz points.

- In general, each peak should be defined by at least 20 data points. For chromatograms with co-eluting peaks or low signal-to-noise ratios, 40 data points per peak is recommended.
- If all peaks are relatively wide, select a slower data collection rate (1.0 Hz, for example).
- If any peaks of interest are less than a few seconds, select a faster data collection rate (5.0 Hz, for example).

- If the data collection rate is too slow, the start and end points of peaks are not accurately determined. If the collection rate is too fast, data files may occupy excessive disk space and post-run analyses may require more processing time.
- A slow data collection ratewith a fast rise time may result in a longer system response than indicated by the rise time. For example, a data collection rate of 0.5 Hz and a rise time of 0.5 second results in a system response longer than 2.0 seconds.
- At a data collection rate of 20 Hz, the 3D sample wavelength range must be ≤ 310 nm. Regardless of the data collection rate, the reference wavelength (if set) must always be within the 3D range.
- The table below lists the recommended pairings for the rise time and data collection rate.

Rise Time (seconds)	Data Collection Rate (Hz)
0.05	20.0
0.1	20.0
0.2	10.0
0.5	5.0
1.0	2.0
2.0	1.0
5.0	0.5

4.3.4 Sample Wavelength

The PDA-3000 measures absorbance over all wavelengths from 190 to 800 nm. The deuterium lamp optimizes the UV range (190 to 380 nm), while the tungsten lamp optimizes the visible range (380 to 800 nm). Set the sample wavelength at the wavelength with the absorbance maxima for the analytes of interest.

When the analyte wavelength maxima are known, the PDA-3000 can run five separate wavelengths without being required to collect spectra. Collecting individual wavelengths instead of the spectra offers two advantages: it uses less disk space and it eliminates the need to perform extractions for analyses that do not require spectral data.

If little is known about the analytes in the sample, collect spectra over the full wavelength range (using both lamps). Although this requires more disk space, it provides complete information about the analytes and the spectra can be used to determine peak identity, purity, and wavelength maxima.

4.3.5 Bandwidth

The bandwidth defines a wavelength range around the wavelength at which the chromatogram is measured. Select a bandwidth that provides the resolution required to achieve the desired sensitivity.

Increasing the bandwidth increases the number of different wavelength signals averaged at each point in time. As the bandwidth increases, baseline noise decreases; however, this may decrease the signal, especially of those peaks that exhibit spectral peak maxima. For narrow spectral peaks, select narrow bandwidths.

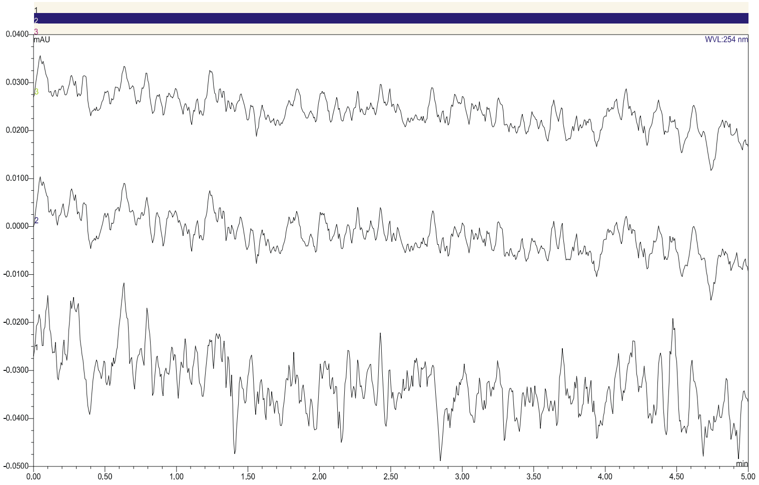
Example Chromatogram: Selecting the Bandwidth

Fig. 26 (→ page 48) illustrates how selecting a wide bandwidth for both the sample and reference wavelengths reduces noise by averaging over a wavelength range. The operating conditions are:

Sample wavelength = 254 nm

Reference wavelength = Off

Flowing water at 1.0 mL/min, 10.35 MPa (1500 psi)



Bandwidth = 8
Noise = 14 µAU

Bandwidth = 4
Noise = 16 µAU

Bandwidth = 1
Noise = 26 µAU

Fig. 26: Effect of Bandwidth on Baseline Noise

4.3.6 Reference Wavelength

The reference wavelength is the center wavelength of the reference intensity determination. Absorbance is measured by comparing the reference intensity to the sample intensity. Selecting a reference wavelength implies a reference mode for each of the single wavelengths being collected. There are two types of reference mode: “active” or “off.”

- To select the active reference mode, enter a wavelength within the selected sample scanning range of the PDA-3000. The selected wavelength will be used as the reference wavelength. The reference intensity will vary over time, as the signal intensity changes.

  For maximum compensation, select a reference wavelength from the same lamp as the sample wavelength. For example:

  - If the sample wavelength is 272 nm, you might select a reference wavelength of 325 nm because both wavelengths are within the UV spectrum (190 to 380 nm) of the deuterium lamp.
  - If the sample wavelength is 572 nm, you might select a reference wavelength of 425 nm because both wavelengths are within the visible spectrum (380 to 800 nm) of the tungsten lamp.

  The active reference mode minimizes baseline drift and is ideal for gradient analyses, where the baseline may shift. This mode also helps compensate for fluctuations in ambient temperature caused by air conditioning.

  NOTE: When the active reference mode is selected, select a reference wavelength and reference bandwidth in a quiet area of the spectrum where little absorption occurs. The reference wavelength plus one-half the reference bandwidth must be within the sample wavelength range selected (→ page 39). If the data collection rate is 20 Hz, the 3D sample wavelength range must be ≤ 310 nm (see page 46).

- To *cancel* the active reference mode, enter “Off” as the reference wavelength. The intensity reading at the time of autozero will be used as the reference intensity.

  When running an isocratic analysis in a laboratory with stable ambient temperature, operating the detector with the active reference mode turned off ensures the lowest baseline noise. It also ensures that fluctuations in signal intensity in the reference wavelength do not cause additional noise.

Example Chromatogram: Selecting the Reference Wavelength

Fig. 27 shows the results obtained from two different channels within the same run. When the reference wavelength mode is “Off” (i.e., the active reference mode is cancelled), the baseline drift is -120 µAU/hour. When the active reference mode is selected, the baseline drift is reduced to -40 µAU/hour.

The operating conditions are:

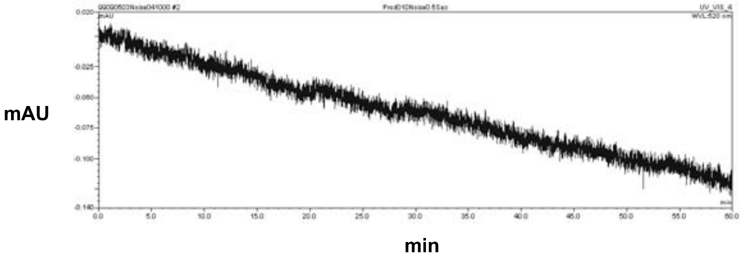
Sample wavelength = 520 nm

Bandwidth = 10 nm

Reference bandwidth = 50 nm (active reference mode only)

Flowing water at 1.0 mL/min, 10.35 MPa (1500 psi)

Reference Wavelength Mode: “Off”
Baseline Drift: -120 μAU/hour



Reference Wavelength Mode: “Active”
Baseline Drift: -40 μAU/hour

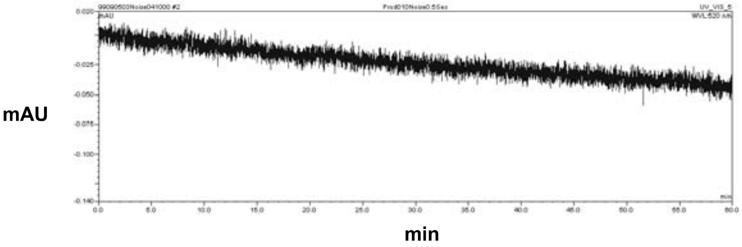


Fig. 27: Effect of Reference Wavelength on Baseline Drift

4.3.7 Reference Bandwidth

The reference bandwidth is analogous to the conventional bandwidth of a channel. The purpose of the reference bandwidth is to average several photodiode signals in a range surrounding the reference wavelength.

It is not necessary to select a reference bandwidth unless you enter a reference wavelength (i.e., you select the active reference mode). If you enter “Off” as the reference wavelength, the reference bandwidth will be the same as the bandwidth.

Select a reference bandwidth that is narrow enough not to interfere with nearby compounds and select an area of the spectrum where the sample does not absorb.

Example Chromatogram: Selecting the Sample Wavelength, Reference Wavelength, and Reference Bandwidth

Fig. 28 (→ page 51) shows the spectrum of caffeine. The operating conditions are:

Sample wavelength = 272 nm
Bandwidth = 5 nm
Reference wavelength = 325 nm
Reference bandwidth = 51 nm
These settings calculate the absorbance by the following equation:

$$\mathrm{AU} = \log \frac{\text{avg } [I_K(300 \text{ to } 350)]}{\text{avg } [I_K(270 \text{ to } 274)]} - \text{offset in AU (at autozero at 272 nm)}$$

Where:

I_K = Light intensity

Offset = Absorbance offset at autozero

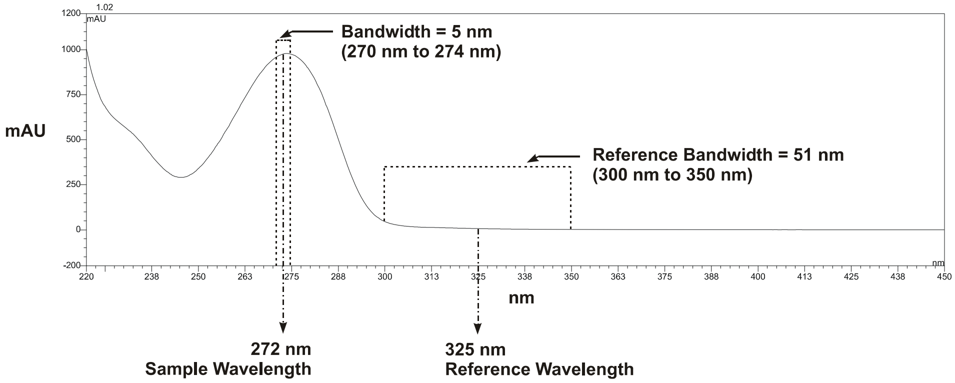


Fig. 28: Caffeine Spectrum

4.3.8 Bunch Width

The bunch width setting determines how many nanometers are averaged when collecting 3D data. The default is 1 nm. Selecting a bunch width above 1 nm will reduce the required data storage by approximately the reciprocal. Do not select a bunch width when the spectrum of the compound has a fine structure; the bunch width will reduce the spectral resolution.

4.3.9 Step

Important: Chromeleon automatically selects the appropriate step setting. Therefore, it is not necessary for users to select a step setting.

A step is the time interval between two successively stored data points. The smaller the step is, the more data points are recorded and, in general, the more precise is the analytical results. However, because collecting more data points requires more storage capacity, the step setting sometimes represents a compromise between the amount of information collected and the file size.

Chromeleon automatically selects the step value that is the inverse of the data collection rate selected by the user. For example, if the data collection rate is 5.0 Hz, the software sets the step to 0.2 second. The following table indicates the step setting automatically selected for each data collection rate.

Data Collection Rate (Hz)	Step (seconds)
0.25	4.0
0.5	2.0
1.0	1.0
2.0	0.5
2.5	0.4
5.0	0.2
10.0	0.1
20.0	0.05

Step Override

Important: This section explains how to override the step setting automatically selected by Chromeleon. This information is provided for reference by advanced users only. Be aware that selecting an inappropriate step setting may cause loss of sensitivity, skipped data points, and other problems.

In almost all cases, the step setting selected by Chromeleon is the preferred setting. However, it is possible to override this setting by manually changing the step in the PGM file or on the Control panel.

For example, you may want to increase the step time for the 3D field or any of the five absorbance channels. For the 3D field, the step determines the sampling rate at which the detector stores spectra. Ideally, the step is no smaller than the interval suggested for the rise time (see the table in the "Data Collection Rate" section on page 46). The advantage of a larger step size is that it reduces the amount of data stored; for example, selecting a step twice as large as that listed in the table reduces the data file size for the 3D field by 50%.

If you select a larger step size when the average parameter is on (see the "Average" section below), the system response is a combination of the selected rise time and the average of the data points between steps. If you select a larger step size when the average parameter is off, the selected rise time is valid, but some data points will be skipped.

4.3.10 Average

The Average parameter operates in conjunction with the Step parameter (see the "Step" section on page 52).

When Average is On, Chromeleon averages the data points between Step intervals, reports that value, and noise is reduced. This setting is recommended for most applications.

When Average is Off, Chromeleon reports the data points at the step interval. The data points between steps are skipped and noise is not reduced.

Important: If the step size is more than double the rise time, the system response is dominated by the step size. The system response time is approximately the larger of (a) the rise time *or* (b) two times the step size (when average is On).

4.3.11 Negative Absorbance

Negative absorbance is the result of decreased baseline absorption of the mobile phase after the autozero routine (which usually occurs at the beginning of a run). At the default setting of 1, the PDA-3000 becomes saturated when the light transmission is 20% above the autozero level. This results in a flat, truncated baseline.

If desired, increase the negative absorbance level in order to continue seeing the actual baseline and peaks. This is especially helpful when running a gradient application in which the absorbance decreases more than 100 mAU. As the following table indicates, the extent of negative absorbance depends on the wavelength. (The values in this table are intended as guidelines only.)

Negative Absorbance Level	Approximate Negative Absorbance at 210 nm	Approximate Negative Absorbance at 240 nm	Approximate Negative Absorbance at 254 nm
0	-2200 mAU	-50 mAU	-300 mAU
1 (default)	-2300 mAU	-100 mAU	-350 mAU
2	-2600 mAU	-200 mAU	-450 mAU
3	-2600 mAU	-300 mAU	-550 mAU
4	-2700 mAU*	-400 mAU	-630 mAU
5	-2700 mAU*	-500 mAU	-660 mAU
6	-2700 mAU*	-600 mAU	-760 mAU
* These signals are saturated and will not report a further negative value.			

1. In Chromeleon, press F8 to display the Commands dialog box.
2. Select **UV** and then select **NegativeAbsorptionLevel**. Enter a new setting and click **Execute**.
3. Select **Autozero** in the Commands dialog box and click **Execute** to run the autozero routine.
4. Select **LampIntensity** in the Commands dialog box to check the intensity of the deuterium lamp. The reading should be above 10 million counts for an analytical or semipreparative cell or above 3 million for a semi-micro cell.

   If the reading is below 10 million counts, select **UV_Calibration Intensity** and click **Execute** to calibrate the intensity of the deuterium lamp. Afterward, check the lamp intensity again. If the reading is still too low, clean the flow cell (→ section 6.2, page 84) or replace the lamp (→ section 6.3, page 91).
5. You may continue increasing the negative absorbance level if the range is required; however, be aware that baseline noise may increase as the negative absorbance increases.

4.4 Wellness and Diagnostics

The PDA-3000 Firmware periodically checks the status of certain system parameters. All problems are reported to the computer and logged in the Chromeleon Audit Trail. The Audit Trail includes the date, time, and severity level for each problem the PDA-3000 reports. There are two ways to review this information:

- Check the Audit Trail displayed in the PDA-3000 Control panel, *or*
- Retrieve the Audit Trail from a timebase by selecting the Audit Trail folder in the timebase and the file for that date in the Chromeleon browser.

Chromeleon includes several diagnostic functions for monitoring the wellness of your chromatography system. These functions help to prevent problems and ensure reliable operation of each module in the system.

- **Lamp age**

Purpose: Indicates the total number of hours that each lamp has been turned on in its lifetime.

To check the number of hours the lamps have been in service:

1. In Chromeleon, press F8 to display the Commands dialog box and select **UV**.
2. Select **UVLampAge**. If the deuterium lamp has been in service for more than 2000 hours, replace it (→ section 6.3.2, page 92).
3. Select **VisLampAge**. If the tungsten lamp has been in service for more than 2000 hours, replace it (→ section 6.3.3, page 95).

To reset the lamp age:

1. After installing a new lamp, press **F8** to display the **Commands** dialog box.
2. Select **UV**.
3. Select the **UVLampAge** command (for the deuterium lamp) or **VisLampAge** command (for the tungsten lamp).
4. Reset the number to zero.
5. Click **Execute**.

NOTE: When the detector power is initially turned on, the lamp age already indicates some elapsed time; this is the time that was required for factory calibration and test procedures.

- **Lamp intensity**

Purpose: Chromeleon provides counters for the UV lamp's intensity at 254 nm in counts per second and for the visible lamp's intensity at 520 nm in counts per seconds. Check the deuterium lamp intensity approximately every 6 months.

Check that at least one lamp is turned on. (When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted.) If necessary, turn on one of the lamps from either Chromeleon or the TTL input.

To check the UV lamp intensity:

1. In Chromeleon, press **F8** to display the **Commands** dialog box and select **UV**.
2. Select **LampIntensity**. The reading should be above 10 million counts for an analytical or semipreparative cell or above 3 million counts for a semi-micro cell. However, a lamp with less intensity may work sufficiently, depending on the noise level required for the application.

If the reading is too low, follow the steps in Section 5.7 (→ page 75) to resolve the problem.

- **Wavelength calibration**

Purpose: Improves wavelength accuracy in the UV range by reassigning new wavelengths to each pixel, based on the emission spectrum from the deuterium lamp. The results of both the initial calibration and the last calibration are stored in the calibration log. You can view the results at any time.

1. Verify the following conditions: no solvent is flowing through the cell, the background absorbance is low, and there are no bubbles in the light path.
2. In the Browser, navigate to the following folder: **Dionex Templates/Panels/Dionex_LC**.
3. Open the **UltiMate_3000_PDA-3000.pan** control panel.
4. Click the **Diagnostics** button. The PDA-3000 diagnostics panel opens. Click the **wavelength** script button under **Calibration**. The detector will perform the wavelength calibration routine and upload the result (pass or fail) to Chromeleon.

If the wavelength calibration fails, follow the steps in Section 5.8 (→ page 76) to resolve the problem.

- **Wavelength verification**

Purpose: Tests the accuracy of the selected wavelength. The result of the last wavelength verification test is stored in the calibration log and can be retrieved at any time. Run the wavelength verification test for both the deuterium and tungsten lamps approximately every 6 months.

1. In the Browser, navigate to the following folder: **Dionex Templates/Panels/Dionex_LC**.
2. Open the **UltiMate_3000_PDA-3000.pan** control panel.
3. Click the **Diagnostics** button. The PDA-3000 diagnostics panel opens. Click the **wavelength verification** script button under **Diagnostic Tests**. The detector will perform the wavelength verification test and upload the result (pass or fail) to Chromeleon.

If the wavelength verification fails, calibrate the wavelength and then run the wavelength verification test again. If the lamp fails again, follow the steps in Section 5.9 (→ page 78) to resolve the problem.

NOTE: After you have performed instrument qualification, perform the **QualificationDone** command to update the **Qualification.LastDate** and **LastOperator** information.

4.5 Stand-Alone Analog Operation

This section describes limited, stand-alone operation of the PDA-3000 when the detector is not interfaced to a computer running Chromeleon. In stand-alone operation, TTL inputs on the PDA-3000 rear panel can be used to trigger autozero or to turn the lamps on and off. All other detector parameters will remain at their factory default settings and cannot be changed.

Factory Default Settings

The PDA-3000 outputs four analog signals at all times, even when the detector is not interfaced to a computer. Data is presented at the analog output channels whenever the detector power is on. A chart recorder or other voltage-measuring device can be used to monitor the channels.

When the PDA-3000 power is first turned on, the analog output settings are at the factory defaults listed in the table below.

Channel	Single Wavelength	Bandwidth	Reference Wavelength	Full-Scale Output	% Offset
1	210	10	Off	1.0 AU	10%
2	254	10	Off	1.0 AU	10%
3	272	10	Off	1.0 AU	10%
4	520	10	Off	1.0 AU	10%

The following default settings are also in effect when the power is first turned on:

- Rise time: 2.0 seconds.
- Lamp status: The deuterium and tungsten lamps are on.

Stand-Alone Operation

The table below indicates which PDA-3000 parameter each TTL input controls during stand-alone operation.

TTL Input	PDA-3000 Parameter
TTL 1 in	Autozero
TTL 2 in	Reserved
TTL 3 in	Deuterium lamp on/off
TTL 4 in	Tungsten lamp on/off

5 Troubleshooting

The following features help you to identify and eliminate the source for problems that may occur during the operation of the detector or an UltiMate 3000 system.

Error Messages

If a fault or mechanical error is detected during the operation of the detector, an error message is displayed in the Chromeleon Audit Trail. Section 5.1 (→ page 60) describes error messages and how to troubleshoot them.

Status Indicators

The status indicators on the front panel display provide a quick visual check of the operational status of the detector. For troubleshooting information when an LED is flashing, refer to section 5.2 (→ page 69).

Operating Problems

Section 5.2 (→ page 69) through Section 5.14 (→ page 81) describe routine operating problems and how to resolve them.

Diagnostics Tests

If the detector is connected in Chromeleon, Chromeleon provides several diagnostic tests allowing you to check the performance of certain detector components. For more information, refer to Section 4.4 (→ page 55).

If you are unable to eliminate a problem following the instructions given here, contact Dionex Service.

5.1 Alarms and Error Conditions

The PDA-3000 Firmware periodically checks the status of certain system parameters. If a problem is detected, it is reported to the Chromeleon PC and displayed in the Chromeleon Audit Trail. Each error message is preceded by an icon that identifies the seriousness of the underlying problem (see the table below). You can change the severity level assigned to a problem whenever appropriate.

Icon	Severity Level	Description
!	Warning	A message is displayed in the Audit Trail, but the current run is not interrupted.
⚠	Error	A message is displayed in the Audit Trail, and the system attempts to correct the problem (sometimes by using an alternative parameter).
Stop	Abort	A message is displayed in the Audit Trail, and the running batch is aborted.

The table below lists the PDA-3000 error messages and their default severity levels. For troubleshooting assistance, refer to the page indicated in the table.

Alarms and Error Conditions	Default Severity Level	See
3DFIELD RefWavelength and/or RefBandwidth is out of range.	Error	page 62
Acquisition On for 3DFIELD rejected--no 3D Data Acquisition icense found.	Abort	page 63
Autozero is already in progress.	Error	page 63
Calibration is running.	Abort	page 63
Cannot change 3DFIELD parameters while data acquisition is running.	Warning	page 63
Cannot set manual parameters for the required operation because the DSP is not ready.	Abort	page 64
Can’t execute calibration command during data acquisition.	Error	page 64
Can’t execute diagnostic command during data acquisition.	Error	page 64
Can’t execute X command during data acquisition.	Error	page 64
Command not executed because an Autozero command is already running.	Warning	page 64
Data is unavailable.	Abort	page 64
Diagnostic is running.	Abort	page 64
DSP communication failure.	Abort	page 65

Alarms and Error Conditions	**Default Severity Level**	**See**
Filter wheel sensor could not detect blocked position.	Warning	page 65
Filter wheel sensor could not detect open position.	Warning	page 65
Invalid rise time. Valid rise times at 20Hz: 0.05, 0.1, 0.2, 0.5, 1.0 and 2.0.	Abort	page 65
Invalid step for the 3DFIELD. Valid steps are 0.05, 0.1, 0.2, 0.4, 0.5, 1, 2, and 4.	Error	page 66
Leak sensor error.	Warning	page 66
Not enough light to perform operation.	Warning	page 66
Over temperature error.	Abort	page 66
PDA is running a calibration or diagnostic function. Not ready to accept this command.	Error	page 67
The minimum wavelength must be less than the maximum wavelength. Min wavelength = X; max wavelength = X.	Error	page 67
The range of wavelength bunching is 1 to 25 nm.	Error	page 67
The range of wavelengths is outside the 190 to 800 nm limit. Wavelength = X; Bandwidth = X.	Error	page 67
UV lamp control circuitry error while lamp is off.	Abort	page 68
UV lamp error or control circuitry error while lamp is on.	Abort	page 67
UV lamp error.	Abort	page 67
UV lamp error while attempting to turn off.	Abort	page 68
UV lamp error while attempting to turn on.	Abort	page 67
UV lamp failed to turn on for the required operation.	Abort	page 67
Visible lamp control circuitry error while lamp is off.	Abort	page 68
Visible lamp error or control circuitry error while lamp is on.	Abort	page 67
Visible lamp error.	Abort	page 67
Visible lamp error while attempting to turn off.	Abort	page 68
Visible lamp error while attempting to turn on.	Abort	page 67
When collecting at 20Hz, the spectrum range cannot be more than 310 nm. Please lower the data rateor narrow the spectrum range.	Abort	page 69

⚠ **3DFIELD RefWavelength and/or RefBandwidth is out of range.**

To troubleshoot:

For all data collection rates other than 20 Hz, select parameters that meet the following criteria:

$$190 \le \lambda \pm \frac{BW}{2} \le 800$$

Where:

λ = Reference wavelength

BW = Reference bandwidth

For example, the following parameters are VALID:

Wavelength = 190 to 800 nm

Reference wavelength = 750 nm

Reference bandwidth = 50 nm

These parameters are valid because 750 nm + 25 nm (50% of 50 nm) = 775 nm, which is less than 800 nm.

The following parameters are NOT VALID:

Wavelength = 190 to 600 nm

Reference wavelength = 750 nm

Reference bandwidth = 50 nm

These parameters are invalid because 750 nm + 25 nm (50% of 50 nm) = 775 nm, which is greater than 600 nm.

When the data collection rate is 20 Hz, select parameters that meet the following criteria:

$$\mathit{Minimum}\,\lambda \le \lambda \pm \frac{BW}{2} \le \mathit{Maximum}\,\lambda$$
$$\mathit{Maximum}\,\lambda - \mathit{Minimum}\,\lambda \le 310\,nm$$

Where:

λ = Reference wavelength

BW = Reference bandwidth

For example, the following parameters are VALID:

Wavelength = 190 to 500 nm

Reference wavelength = 450 nm

Reference bandwidth = 50 nm

These parameters are valid because 190 to 500 nm is a range of ≤ 310 nm and 450 nm + 25 nm (50% of 50 nm) = 475 nm, which is less than 500 nm.

The following parameters are NOT VALID:

Wavelength = 190 to 600 nm

Reference wavelength = 750 nm

Reference bandwidth = 50 nm

These parameters are invalid because 190 to 600 nm is a range of more than 310 nm; also, 750 nm + 25 nm (50% of 50 nm) = 775 nm, which is greater than 600 nm.

Stop **Acquisition On for 3DFIELD rejected--no 3D Data Acquisition license found.**

To troubleshoot:

Chromeleon cannot perform digital data acquisition of a 3D data field unless the software includes the 3D Data Acquisiton license. Contact Dionex if you want to order this option.

⚠ **Autozero is already in progress.**

To troubleshoot:

This message appears if you select Autozero (in the Commands dialog box) when the autozero routine is already in progress. Allow the autozero routine to continue running until completion.

Stop **Calibration is running.**

To troubleshoot:

This message appears if you attempt to issue a command or select a parameter while a calibration procedure is in progress. Wait until the procedure finishes running before making any changes.

❗ **Cannot change 3DFIELD parameters while data acquisition is running.**

To troubleshoot:

This message appears if you attempt to select a parameter for the 3D field while data acquisition is in progress. Wait until data acquisition is complete, or stop the run and then change the parameters.

🛑 **Cannot set manual parameters for the required operation because the DSP is not ready.**

To troubleshoot:

This message appears if certain commands are issued before the DSP (digital signal processor) is ready to accept them. Turn off the PDA-3000 power for 30 seconds, and then turn it on again. If the error message appears again, contact Dionex Service for assistance.

❗ **Can't execute calibration command during data acquisition.**

⚠ **Can't execute diagnostic command during data acquisition.**

⚠ **Can't execute X command during data acquisition.**

To troubleshoot:

During data acquisition, no other commands can be executed. Wait until data acquisition is completed before attempting to issue any other command.

❗ **Command not executed because an Autozero command is already running.**

To troubleshoot:

Wait until the autozero routine is completed before attempting to issue any other command.

🛑 **Data is unavailable.**

To troubleshoot:

This message appears if the PDA-3000 fails to respond when the computer attempts to communicate with the detector. Shut down the Chromeleon server and turn off the PDA-3000 power; wait 30 seconds, and then power up both the server and the detector. If the error message appears again, contact Dionex Service for assistance.

🛑 **Diagnostic is running.**

To troubleshoot:

This message appears if you attempt to issue a command or select a parameter while a diagnostic routine is running. Wait until the diagnostic routine is completed before making any changes.

DSP communication failure.

To troubleshoot:

This message appears if there is a communication failure between the DSP (digital signal processor) and the CPU. Turn off the PDA-3000 power for 30 seconds, and then turn it on again. If the error message appears again, contact Dionex Service for assistance.

Filter wheel sensor could not detect blocked position.

Filter wheel sensor could not detect open position.

A backup paddle alignment routine ensures that the PDA-3000 remains functional even if the sensor cannot detect the filter wheel position. Thus, if this is the only error reported, you may continue operation and defer troubleshooting, if desired.

To troubleshoot:

1. Turn off the PDA-3000 power for 30 seconds, and then turn it on again.
2. If the error message appears again, check the deuterium lamp intensity (→ page 55). The intensity reading should be above 10 million counts for an analytical or semipreparative cell *or* above 3 million counts for a semi-micro cell. If the reading is acceptable, resume normal operation. If the reading is too low, see section 5.7 (→ page 75).
3. If the error message appears again, there may be a faulty connection to the filter wheel sensor. Contact Dionex Service for assistance.

NOTE: The PDA-3000 electronics cannot be serviced by users. All repairs of the electronics components must be performed by a Dionex Service Representative.

Invalid rise time. Valid rise times at 20 Hz: 0.05, 0.1, 0.2, 0.5, 1.0, and 2.0.

To troubleshoot:

This message appears if you enter a rise time setting that is invalid for a data collection rate of 20 Hz. Select a valid rise time.

⚠ **Invalid step for the 3DFIELD. Valid steps are 0.05, 0.1, 0.2, 0.4, 0.5, 1, 2, and 4.**

To troubleshoot:

This message appears if you enter an invalid step setting. Chromeleon automatically selects the step value that is the inverse of the data collection rate. In future, accept the value selected by the software.

Important: Only advanced users should override the step setting selected by Chromeleon. Selecting an inappropriate step setting may cause loss of sensitivity, skipped data points, and other problems.

❗ **Leak sensor error.**

To troubleshoot:

Locate the source of the leak. First, tighten (or replace) all liquid line connections.

If the cell is leaking, check that the backpressure on the cell does not exceed the specification (→ section 6.2.6, page 91).

Wipe up all liquid and dry the leak sensor before resuming operation.

If the cell leaks after you resume operation, replace it (→ section 6.2.4, page 87).

❗ **Not enough light to perform operation.**

To troubleshoot:

Follow the troubleshooting steps in Section 5.7 (→ page 75).

🛑 **Over temperature error.**

To troubleshoot:

The fan that cools the optical bench may have malfunctioned or a component on the lamp supply board may have failed. Turn off the PDA-3000 power and contact Dionex Service for assistance.

NOTE: Do not attempt to service the PDA-3000 electronics components. All repairs of the electronics components must be performed by a Dionex Service Representative.

PDA is running a calibration or diagnostic function. Not ready to accept this command.

To troubleshoot:

Wait until the calibration procedure or diagnostic routine finishes running before attempting to issue any command.

The minimum wavelength must be less than the maximum wavelength. Min wavelength = X; max wavelength = X.

To troubleshoot:

Select a minimum wavelength that is less than the maximum wavelength. Both wavelengths must be within the 190 to 800 nm range.

The range of wavelength bunching is 1 to 25 nm.

To troubleshoot:

This message appears if you enter an invalid setting for the bunch width. Select a bunch width setting from 1 to 25 nm.

The range of wavelengths is outside the 190 to 800 nm limit.

To troubleshoot:

The PDA-3000 can measure the absorbance spectrum from 190 to 800 nm. Make sure both the minimum and maximum wavelengths are within this range.

UV lamp error.

UV lamp error or control circuitry error while lamp is on.

UV lamp failed to turn on for the required operation.

UV lamp error while attempting to turn on.

Visible lamp error.

Visible lamp error or control circuitry error while lamp is on.

Visible lamp error while attempting to turn on.

Visible lamp failed to turn on for the required operation.

To troubleshoot:

1. Check that the lamp referred to in the error message is turned on. When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted. If necessary, turn on the lamp from either Chromeleon or the TTL input.
2. If you recently installed a new deuterium lamp, check that the UV lamp connector wires are routed through the wire guide in the optical bench (→ Fig. 35, page 93) and are not pinched under the lamp cover.
3. Check the number of hours the lamp has been in service (→ page 55)

   If the deuterium lamp has been in operation for *more* than 2000 hours, replace it (→ section 6.3, page 91). If the error message appears again, contact Dionex Service for assistance.

   If the deuterium lamp has been in operation for *less* than 2000 hours, turn off the lamp for at least 3 minutes and then turn it on again. If the error message reappears, replace the lamp (→ section 6.3, page 91). If the error message does *not* appear again, run a manual data acquisition for about 30 minutes. If you notice a shift in the absorbance signal during this time, replace the lamp.

   If the tungsten lamp has been in operation for *more* than 2000 hours, replace it (→ section 1, page 92). If the error message appears again, contact Dionex Service for assistance.

   If the tungsten lamp has been in operation for *less* than 2000 hours, turn off the lamp for at least 30 seconds and then turn it on again. If the error message reappears, replace the lamp (→ section 1, page 92). If the error message does *not* appear again, run a manual data acquisition for about 30 minutes. If you notice a shift in the absorbance signal during this time, replace the lamp.

Stop **UV lamp control circuitry error while lamp is off.**

Stop **UV lamp error while attempting to turn off.**

Stop **Visible lamp control circuitry error while lamp is off.**

Stop **Visible lamp error while attempting to turn off.**

To troubleshoot:

The lamp power supply may have failed or the CPU board may have failed. Contact Dionex Service for assistance.

NOTE: The PDA-3000 electronics cannot be serviced by users. All repairs of the electronics components must be performed by a Dionex Service Representative.

When collecting at 20 Hz, the spectrum range cannot be more than 310 nm. Please lower the data rate or narrow the spectrum range.

To troubleshoot:

Reduce the data collection rate to less than 20 Hz or select a wavelength range that is ≤ 310 nm.

5.2 Flashing LED

- **Run LED is flashing**

  The leak sensor in the drip tray may have been triggered. Check the Chromeleon Audit Trail for a leak-related error message. Find and eliminate the source of the leak.

- **Deuterium LED is flashing**

  The lamp did not power on properly. Check the Chromeleon Audit Trail for a lamp-related error message. If necessary, replace the lamp (→ section 6.3, page 91).

  If the LED flashes again, there may be a bad connection between the lamp power supply and the lamp. Contact Dionex Service for assistance.

  NOTE: The PDA-3000 electronics cannot be serviced by users. All repairs of the electronics components must be performed by a Dionex Service Representative.

- **Tungsten LED is flashing**

  The lamp did not power on properly. Check the Chromeleon Audit Trail for a lamp-related error message. If necessary, replace the lamp (→ section 1, page 92).

  If the LED flashes again, there may be a bad connection between the lamp power supply and the lamp. Contact Dionex Service for assistance.

  NOTE: The PDA-3000 electronics cannot be serviced by users. All repairs of the electronics components must be performed by a Dionex Service Representative.

- **Power LED is flashing**

  If the **Power** LED starts flashing the first time the deuterium lamp is turned on, the detector failed the start-up routine. Follow the steps below to resolve the problem.

  1. Remove the cell from the optical bench (→ section 6.2.4, page).
  2. In Chromeleon, press F8 to display the Commands dialog box and select **UV**.

3. Select **Autozero** and click **Execute** to run the autozero routine. If this corrects the problem, the **Power** LED will stop flashing and you may replace the cell in the optical bench and resume normal operation. If the **Power** LED continues flashing, go on to step 4.
4. Check the deuterium lamp intensity (→ page 55). The intensity reading should be above 10 million counts for an analytical or semipreparative cell *or* above 3 million counts for a semi-micro cell. If the reading is acceptable *and* the **Power** LED is no longer flashing, resume normal operation. If the reading is acceptable but the **Power** LED is still flashing, contact Dionex Service for assistance. If the reading is too low, follow the troubleshooting steps in section 5.7 (→ page 75).

5.3 Lamp Does Not Light or Lamp LED Is Flashing

- **Lamp default setting is not in effect**

When the factory default settings are in effect, the deuterium and tungsten lamps turn on automatically each time the detector power is turned on. If the required lamp does not turn on automatically, you can turn it on from Chromeleon or from the TTL input.

To restore the default setting for a lamp:

1. Press F8 in Chromeleon to display the Commands dialog box and select **UV**.
2. Select **UVLampPowerOnSetting** (for the deuterium lamp) and click **Execute**.
3. Select **VisLampPowerOnSetting** (for the tungsten lamp) and click **Execute**.

- **Lamp is old or burned out**

Replace the lamp. See section 6.3 (→ page 91) or section 1 (→ page 92) for instructions on replacing the deuterium lamp or tungsten lamp, respectively.

5.4 No Detector Response

- **Detector power not on**

Check that the power cord is connected from the PDA-3000 rear panel to a power source.

Check that the main **Power** switch is turned on.

Check the fuses and replace them, if necessary (→ section 6.4,page 95).

- **Lamp not turned on**

Turn on the lamp from either Chromeleon or the TTL input. When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted.

- **Lamp LED flashing**

  The corresponding lamp failed to power on properly. In Chromeleon, press F8 to display the Commands dialog box and select **UV**. Check that the command (**UVLampPowerOnSetting** or **VisLampPowerOnSetting**) is **On**.

  If the LED continues flashing, replace the lamp. See section 6.3 (→ page 91) or section 1 (→ page 92) for instructions on replacing the deuterium lamp or tungsten lamp, respectively.

- **Detector and/or Control panel not connected to timebase**

  In the Chromeleon Server Configuration program, add the PDA-3000 driver to a timebase. For details, refer to the section on the Server Configuration Program in the Chromeleon Administrator Help.

- **Windows operating system does not recognize PDA-3000**

  When the computer is turned on, Windows scans the network for unknown devices. If the PDA-3000 power is off, the detector cannot be identified.

  If you connect the PDA-3000 to the computer before installing Chromeleon, a Windows message box requesting a USB configuration file (cmwdmusb.inf) will appear when you turn on the computer. See Section 5.14 (→ page 81) for instructions on how to resolve this.

5.5 Noisy Baseline

- **Mobile phase or post-column reagent contains light-absorbing impurities**

  Prepare all mobile phases and reagents with spectro-grade solvents, reagent-grade chemicals, and ASTM Type I (or better) filtered, DI water.

- **Leaking fittings**

  Locate the source of the leak. Tighten (or replace) all liquid line connections.

- **Insufficient time for system equilibration after turning on lamp(s) or after changing mobile phase strength or composition**

  The lamps usually require 20 to 30 minutesto stabilize. For sensitive applications work or for detector validation, allow at least 2 hours for the lamps to stabilize. After installing a new lamp, allow from 8 to 24 hours for the lamp to stabilize.

  After changing mobile phases, allow 20 to 30 column volumes for the system to stabilize.

- **Strongly-retained components from previous analysis are slowly eluting**

  To elute strongly-retained species, use a stronger mobile phase. Re-equilibrate with the standard mobile phase before resuming routine operation.

  Clean the column as instructed in the column manual. If the problem persists, refer to the column manual for troubleshooting guidance.

- **Plot scale exaggerates baseline noise**

  If autoscale is selected, even a plot with low noise may fill the screen. Check the Y-axis scale values.

- **Air bubbles in flow cell**

  Remove the trapped air (→ section 6.2.3, page 86).

  To prevent air from becoming trapped in the cell again, follow these steps:

  - Degas mobile phases and post-column reagents by vacuum degassing or sparging with helium.
  - Connect a backpressure line to the cell (→ section 3.6, page 29). This increases the backpressure on the cell, thereby shrinking bubbles and allowing them to pass more easily through the cell.

- **Contaminants in flow cell**

  Clean the cell (→ section 6.2, page 84).

- **Incorrect lamp selected**

  If a UV wavelength is selected, make sure the deuterium lamp is on. If a visible wavelength is selected, make sure the tungsten lamp is on. When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted.

- **Lamp needs replacement**

  The light output of the lamp decreases over time. The lower light output may increase noise above an acceptable level, especially if the lamp has been in service for more than 2000 hours. If this occurs, replace the lamp. See Section 6.3 (→ page 91) or Section 1 (→ page 92) for instructions on replacing the deuterium lamp or tungsten lamp, respectively.

- **Lamp incorrectly installed**

  Make sure that both lamps are fully seated in the optical bench. See Section 6.3 (→ page 91) or Section 1 (→ page 92) for installation instructions for the deuterium lamp or tungsten lamp, respectively.

- **Inappropriate reference wavelength**

  Select a reference wavelength in a quiet area of the spectrum where little absorption occurs. Dionex recommends selecting a reference wavelength below 380 nm for UV applications and above 600 nm for visible applications. For more information, see page 49.

- **Bandwidth is too narrow**

  Select a wider bandwidth and reference bandwidth, if appropriate for the application. For guidelines when selecting the sample and reference bandwidths, see page 47 and page 49.

- **Front panel, lamp cover, or flow cell cover is missing**

  Optical components are sensitive to temperature and light. Do not operate the PDA-3000 unless the front panel, lamp cover, and flow cell cover are in place.

- **Crimped or plugged tubing**

  Over time, tubings may become pinched off and plugged. Periodically check all tubing and replace as needed (→ section 6.1, page 83).

- **Flow cell installed incorrectly**

  Check that the cell is inserted straight into the optical bench and is fully seated.

- **Inappropriate rise time**

  In general, the rise time should be approximately 25% of the peak width at half-height of the narrowest peak of interest. See page 45 for more information.

- **Detector exposed to high vibration**

  Optical detectors are sensitive to vibrations. Make sure the PDA-3000 installation site is vibration-free.

- **Filter paddle has malfunctioned**

  If you have recently autozeroed the detector, check the Audit Trail. If the "Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see page 65 for instructions.

- **Pump needs priming and/or maintenance**

  If the baseline noise is synchronized with the pump stroke, reprime the pump and clean the piston seals and/or check valves. Refer to the pump user's manual for instructions.

- **Negative absorbance level is too high**

  Baseline noise may increase as the negative absorbance increases. If the noise is unacceptable, reduce the negative absorbance level (→ page 53).

5.6 Drifting Baseline

- **Fluctuations in ambient temperature**

  Make sure the PDA-3000 installation site temperature remains consistent. Allow at least 6 cm (2.4 in) of clearance behind the PDA-3000 for ventilation.

  Check that the flow cell inlet tubing is routed through the heat exchanger (→ Fig. 6, page 16) and is correctly connected to the inside front panel (→ Fig. 31, page 89). Check that solvent flow is directed to the cell inlet and out the cell outlet.

  Select the active reference mode by entering a reference wavelength (→ page 49).

- **Front panel, lamp cover, or flow cell cover is missing**

  Optical components are sensitive to temperature and light. Do not operate the PDA-3000 unless the front panel, lamp cover, and flow cell cover are in place.

- **Flow cell heat exchanger not in place**

  Check that the flow cell inlet tubing is threaded through the tubing guide on the lamp cover and secured in place with the tubing bracket (→ Fig. 30, page 88).

- **Lamp not stabilized or burned in**

  The lamps usually require 20 to 30 minutes to stabilize. For sensitive applications work or for detector validation, allow at least 2 hours for the lamps to stabilize. After installing a new lamp, allow from 8 to 24 hours for the lamp to stabilize.

  After changing mobile phases, allow 20 to 30 column volumes for the system to stabilize.

- **Leaking flow cell**

  Tighten fittings. Also, check that the backpressure on the cell does not exceed the specification (→ section 6.2.6, page 91).

  If the cell continues to leak, replace it (→ section 6.2.4, page 87).

- **Absorbance in the reference wavelength**

  Sample absorbance in the selected reference wavelength may cause excessive baseline drift. Select a reference wavelength and reference bandwidth in an area of the spectrum where little absorption occurs (→ page 50).

  Dionex recommends selecting a reference wavelength (i.e., active reference mode) for gradient analyses and turning off the reference wavelength for isocratic analyses in a laboratory with a stable ambient temperature (→ page 49).

5.7 Deuterium Lamp Intensity Reading Too Low

- **Light signal is not reaching the data processor**

  Check that at least one lamp is turned on. (When a lamp is on, the corresponding front panel LED (**Deuterium** or **Tungsten**) is lighted.) If necessary, turn on one of the lamps from either Chromeleon or the TTL input.

- **Low light throughput**

  1. Remove the cell from the optical bench (→ section 6.2.4, page 87).
  2. Check the deuterium lamp intensity (→ page 55).

     If the intensity reading is above 10 million counts for an analytical or semipreparative cell *or* above 3 million counts for a semi-micro cell, follow these steps:

     a. In Chromeleon, press F8 to display the Commands dialog box.
     b. Select **UV** and **UV_Calibration**.
     c. Select **Intensity** and click **Execute** to calibrate the lamp intensity.
     d. Select **Autozero** (in the Commands dialog box) and click **Execute** to run the autozero routine.
     e. If no error message is displayed, flush the cell with the mobile phase for the application and/or clean the cell as instructed in Section 6.2 (→ page 84).
     f. Reinstall the cell in the optical bench.

g. Autozero the detector again to verify that the problem has been resolved. If the problem remains, contact Dionex Service for assistance.

If the intensity reading is below 10 million counts for an analytical or semipreparative cell *or* below 3 million counts for a semi-micro cell, follow these steps:

a. Check that the flange of the deuterium lamp is aligned with the two locating pins in the optical bench (→ Fig. 35, page 93). If the flange is aligned, the lamp is defective and should be replaced (→ section 6.3, page 91).

b. If the intensity reading is still too low after the lamp was replaced, contact Dionex Service for assistance.

- **Filter paddle has malfunctioned**

  Check the Audit Trail. If the Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see page 65 for instructions.

- **Failure of an electronics component**

  Turn off the PDA-3000 power and contact Dionex Service for assistance.

  NOTE: Do not attempt to service the PDA-3000 electronics components. All repairs of the electronics components must be performed by a Dionex Service Representative.

5.8 Wavelength Calibration Fails

- **Deuterium lamp may be defective**

  If the **Deuterium** LED is flashing—or if the LED fails to light—replace the deuterium lamp (→ section 6.2.4, page 87).

  If the **Deuterium** LED is lighted but is not flashing, open the PDA-3000 Wellness panel and run the wavelength calibration and the wavelength verification test. If the PDA-3000 fails these procedures, remove the flow cell from the detector and repeat both procedures. If the detector fails the wavelength calibration again, contact Dionex Service for assistance.

- **Deuterium lamp intensity out of calibration**

  1. Remove the cell from the optical bench (→ section 6.2.4, page 87).
  2. Check the deuterium lamp intensity (→ page 55).

     If the intensity reading is above 10 million counts for an analytical or semipreparative cell *or* above 3 million counts for a semi-micro cell, follow these steps:

     a. In Chromeleon, press F8 to display the Commands dialog box.
     b. Select **UV** and **UV_Calibration**.
     c. Select **Intensity** and click **Execute** to calibrate the lamp intensity.
     d. Select **Autozero** (in the Commands dialog box) and click **Execute** to run the autozero routine.
     e. If no error message is displayed, flush the cell with the mobile phase for the application and/or clean the cell as instructed in Section 6.2 (→ page 84).
     f. Reinstall the cell in the optical bench.
     g. Autozero the detector again to verify that the problem has been resolved. If the error message appears again, contact Dionex Service for assistance.

     If the intensity reading is below 10 million counts for an analytical or semipreparative cell *or* below 3 million counts for a semi-micro cell, follow these steps:

     a. Check that the flange of the deuterium lamp is aligned with the two locating pins in the optical bench (→ Fig. 35, page 93). If the flange is aligned, the lamp is defective and should be replaced (→ section 6.3, page 91).
     b. If the error message appears again, contact Dionex Service for assistance.

- **Flow cell contains air bubble or old mobile phase**

  Flush the cell with the mobile phase for the application.

- **Cell not clear**

  Clean the cell (→ section 6.2, page 84).

  If the detector fails the wavelength calibration again, replace the flow cell windows (→ section 6.2.5, page 89). Contaminated flow cell windows can significantly increase baseline noise and cause wavelength calibration to fail.

5.9 Wavelength Verification Fails

- **Detector wavelength requires recalibration**

  Open the PDA-3000 Wellness panel and run the wavelength calibration procedure (→ page 56).

- **No solvent flow through cell**

  If the **Power** LED is flashing (→Fig. 3, page 11), flush the cell with DI water or methanol and then turn off the detector power for 30 seconds. Turn on the power again and verify that the **Power** LED is no longer flashing.

  Check the Audit Trail. If the "Not enough light to perform operation." message is displayed, flush the cell with DI water or methanol until the autozero routine can be performed without error.

- **Lamp(s) needs replacement**

  Check the number of hours the lamps have been in service (→ page 55) and replace them if necessary.

- **Solvent absorbs at the wavelength reported for verification**

  Pump DI water or methanol through the system during the wavelength verification test.

- **Filter paddle position error**

  Check the Audit Trail. If the "Filter wheel sensor could not detect blocked position." or "Filter wheel sensor could not detect open position." message is displayed, see page 65 for instructions.

5.10 No Spectra Collected

- **Spectral range was not programmed**

  In Chromeleon, select a spectral range in the PGM file or on the PDA-3000 Control panel.

5.11 Low Spectral Resolution

- **Bunch width too wide**

  Select a bunch width setting of 1 nm, and then check the resolution again.

- **Incorrect reference wavelength**

  Select a reference wavelength where minimum absorbance occurs. Make sure the reference wavelength is included in the wavelength range.

- **Wavelength range too narrow**

  Make sure the spectrum wavelength range includes both the reference wavelength and reference bandwidth. Make sure the peak absorbance wavelength is not included in the reference bandwidth.

- **Reference bandwidth too wide**

  Select a narrower reference bandwidth. The reference bandwidth should not overlap the bandwidth.

5.12 Peaks Too Large or Small

- **Analog data collection**

  Check the recorder and integrator input voltage. The PDA-3000 provides a 1 volt full-scale output.

  Verify that the selected output range (AU full-scale) is appropriate.

- **System-level problem**

  Verify that the sample volume or concentration is appropriate.

  Verify that the rise time (→ page 45) and data collection rate (→ page 46) are appropriate to capture the narrowest peak.

  Prepare fresh post-column reagent.

  Verify that the reagent flow rate is correct.

- **Reference bandwidth too wide**

  Select a narrower reference bandwidth. The reference bandwidth should not overlap the sample bandwidth.

5.13 Poor Peak Shape

- **Concentration of standard is too high; column is overloaded**

  Dilute the standard.

  Install a smaller sample loop or inject a smaller volume.

- **Concentration of standard is too low**

  Increase the standard concentration.

- **Inappropriate mobile phase**

  Do not use a mobile phase that absorbs strongly at the peak absorbance wavelength.

- **Incorrect sample wavelength selected**

  If little is known about the analytes in the sample, collect spectra over the full wavelength range (using both lamps). For guidelines to follow when selecting the sample wavelength, see page 47.

- **Incorrect reference wavelength selected**

  Select a different reference wavelength. For guidelines to follow when selecting the reference wavelength, see page 49.

- **Bandwidth too wide**

  Select a bandwidth that provides the resolution required to achieve the desired sensitivity. Narrow spectra peaks require narrow bandwidths. A wider bandwidth reduces baseline noise, but may reduce peak height.

- **Reference bandwidth too wide**

  Select a bandwidth that provides the resolution required to achieve the desired sensitivity. A wider bandwidth reduces baseline noise, but may increase the chance that the sample absorbs in the reference bandwidth, thus causing reduced sensitivity and peak height.

- **Cell flow is reversed**

  Check that the flow cell inlet tubing is routed through the heat exchanger (→ Fig. 6, page 16) and is correctly connected to the inside front panel (→ Fig. 31, page 89). Check that solvent flow is directed to the cell inlet and out the cell outlet.

- **Semi-micro cell flow rate selected when analytical cell is installed**

  For operation at less than 1.0 mL/min, install a semi-micro flow cell.

5.14 Faulty USB Communication

- **PDA-3000 not recognized by Windows operating system**

  Dionex strongly recommends installing Chromeleon *before* connecting the PDA-3000 to the computer. When the software is installed first, USB driver information is loaded automatically.

  If you connect the PDA-3000 to the computer before installing Chromeleon, a Windows message box requesting a USB configuration file (cmwdmusb.inf) will appear when you turn on the computer. Follow these steps to resolve the problem:

  1. Click **Cancel** in the Windows message box.
  2. Turn off the power to the PDA-3000 and unplug the USB cable from the computer.
  3. Install Chromeleon.
  4. Reconnect the USB cable to the computer and turn on the power to the PDA-3000. Windows will now automatically recognize the detector.

6 Routine Maintenance

This chapter describes routine service procedures that users may perform. All procedures not described here must be performed by Dionex Service personnel.

NOTE: All repairs of the PDA-3000 electronics must be performed by qualified Dionex Service personnel.

Before replacing any parts, review the troubleshooting information in Chapter 5 (→ page 59) to isolate the cause of the problem.

Important: Substituting non-Dionex parts may impair the performance of the PDA-3000, thereby voiding the product warranty. Refer to the warranty statement in the terms of sale for more information.

Important: Before you return any instrument to Dionex for repair, contact Dionex Service or your local distributor. An RMA number (Return Material Authorization number) is required in order to track your instrument. Always use the original packaging and observe the packing instructions (→ Service Return Form section in this manual binder) when shipping the PDA-3000. Shipping the instrument in anything other than the original packaging will void the warranty. Refer to the warranty statement in the terms of sale for more information.

6.1 Regular Maintenance Procedures

- Before shutting down the detector for more than 24 hours, flush the system with deionized (DI) water or methanol for at least 15 minutes at 1.0 mL/min (or maintain a continuous flow at 0.2 mL/min). This rinses corrosive acids, salts, or bases from the flow paths.
- Periodically check the waste line connected to the leak tray at the bottom of the optical bench. Verify that the tubing is unclogged and that it is routed below the leak tray.
- Periodically check all tubing and fittings and replace as necessary (→ section 4.4, page 55) Tubing and fitting components may eventually become loose and leak, or pinched off and plugged. Look for these symptoms:

  Decreasing pressure; this may indicate a leak.

  A sudden increase in backpressure; this may indicate plugged tubing. (A slow increase in backpressure is normal as the separator column ages.) To troubleshoot, bypass the column and then start the pump; the backpressure should decrease substantially.

Without the column, backpressure is usually less than 0.34 MPa (50 psi), depending on the flow rate and tubing ID. Note that the semi-micro flow cell will develop approximately 1.03 MPa (150 psi) backpressure at 1.0 mL/min.

If the backpressure remains high, remove the fittings and tubing from part of the flow path, section by section; starting at the outlet connection, work back "upstream" until you identify the section causing the substantial backpressure. Replace tubing and fittings as necessary.

- Periodically check the flow cell tubing connections for leaks or restrictions. Replace tubing and fittings as necessary.
- Periodically repassivate stainless steel systems. This is especially important when operating with extreme pH levels (which will shorten the life of the passivation finish) or after a major disassembly.
- Monitor baseline noise. If the noise increases above an acceptable level, especially after a lamp(s) has been in service for an extended length of time, replace the lamp (→ section 6.3, page 91).
- Use the diagnostic functions in Chromeleon to check the lamp lifetime, intensity, and wavelength calibration (→ section 4.4, page 55).

6.2 Flow Cell

The table below lists the flow cells available for use with the PDA-3000. All cells are shipped with outlet fluidics, backpressure line and capillary to the column compartment and fittings.

Description	Part No.
Analytical flow cell (cell volume: 13 µl, cell material: PEEK)	6080.0200
Analytical flow cell (cell volume: 13 µl, cell material: steel)	6080.0210
Semi-micro flow cell (cell volume: 3.1 µl, cell material: PEEK)	6080.0220
Semi-micro flow cell (cell volume: 3.1 µl, cell material: steel)	6080.0230
Semi-preparative flow cell (cell volume: 0.7 µl, cell material:PEEK)	6080.0240

The following components are available as replacement parts:

Description	PEEK P/N.	Stainless Steel P/N
Reducing union fittings (analytical and semi-micro cells)	6080.2101	6080.2102
Union fittings (semipreparative cell)	6080.2103	-
Cell outlet tubing (analytical and semi-micro cells)	6080.2401 0.38-mm (0.015-in) ID	6080.2403 0.25-mm (0.010-in) ID
Cell inlet or outlet tubing (semipreparative cell)	6080.2406 0.51-mm (0.020-in) ID	-
Outlet fluidics kit (analytical cell), including capillary with tubing and fitting	6080.2410	
Outlet fluidics kit (semi-micro cell), including capillary with tubing and fitting	6080.2411	
Outlet fluidics kit (semipreparative cell), including capillary with tubing and fitting	6080.2412	

6.2.1 Liquid Leaks

NOTE: Periodically check all tubing and fittings and replace as necessary (→ section 4.4, page 55) Tubing and fitting components may eventually become loose and leak, or pinched off and plugged.

The inlet tubing of the analytical and semi-micro cells cannot be replaced. If a leak (at the flow cell inlet, for example) cannot be eliminated by tightening the fitting, replace the entire cell (→ section 6.2.4, page 87).

If you have located the source of a leak, tighten (or replace) all liquid line connections first.

If the cell is leaking, check that the backpressure on the cell does not exceed the specification (→ section 6.2.6, page 91).

Wipe up all liquid and dry the leak sensor before resuming operation.

If the cell leaks after you resume operation, replace it (→ section 6.2.4, page 87).

6.2.2 Cleaning the Flow Cell

Film deposits on the flow cell windows may cause excessive baseline noise or high absorbance offset.

1. Pump methanol through the cell for 20 to 30 minutes at 1.0 mL/min.
2. Pump DI (deionized) water through the cell for 20 to 30 minutes at 1.0 mL/min.

If the procedure above does not clean the cell, follow these steps:

1. Disconnect the liquid lines from the cell in and out connections.
2. Connect a luer adapter to the cell in connection.
3. Using a syringe, flush the cell with a succession of at least 10 µL each of DI water, acetone, and 3 M HNO_3.
4. Reconnect the cell liquid lines.
5. Flush the cell with DI water for 20 to 30 minutes at 1.0 mL/min.

6.2.3 Removing Trapped Air from the Flow Cell

Trapped air bubbles in the flow cell may cause pulsations of the baseline or random noise and low readings.

Flush the cell with methanol for 20 minutes. If this does not remove the trapped air, follow these steps:

1. Disconnect the liquid line and/or backpressure device from the cell out connection.
2. Using a small syringe, push 3 to 5 mL of methanol through the cell. Or, bypass the column and pump methanol through the system at 1.0 mL/min.
3. Reconnect the liquid line and/or backpressure device.

6.2.4 Replacing the Flow Cell

The detector is shipped without a flow cell. Before initial operation of the detector, install a flow cell that is appropriate for the respective application.

NOTE: No tools are required for removing and installing the flow cell cover and tubing bracket. The knurled head screws can be loosened and tightened by hand.

Important: Make sure not to twist the end of the flow cell inlet and outlet tubings when connecting them from /to the liquid line connections. Preferably use a tool to hold the union fitting while you remove or install any connections.

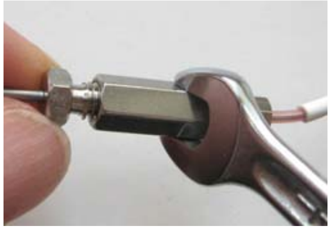


Fig. 29: Union fitting

Removing the Flow Cell from the Optical Bench

Important: When the flow cell is removed, light may emit from the opening to the right of the flow cell. Looking directly into the light source. will cause immediate injury to your eyes.

Remove the flow cell from the optical bench before replacing the cell windows (→ section 6.2.5), installing a new cell, or inspecting the cell for leaks or obstructions.

1. Open the front panel door.
2. Disconnect the tubing connected to the flow cell inlet and outlet.
3. Remove the two knurled head screws on the flow cell cover, and then remove the cover. (→Fig. 4, page 13).
4. **Analytical or semi-micro cell:** Remove the two knurled head screws on the tubing bracket to remove the bracket from the lamp cover (→Fig. 30).
5. Remove the flow cell inlet tubing from the tubing guide in the lamp cover (→ Fig. 30).

6. Squeeze the handle of the flow cell and pull it out of the optical bench.

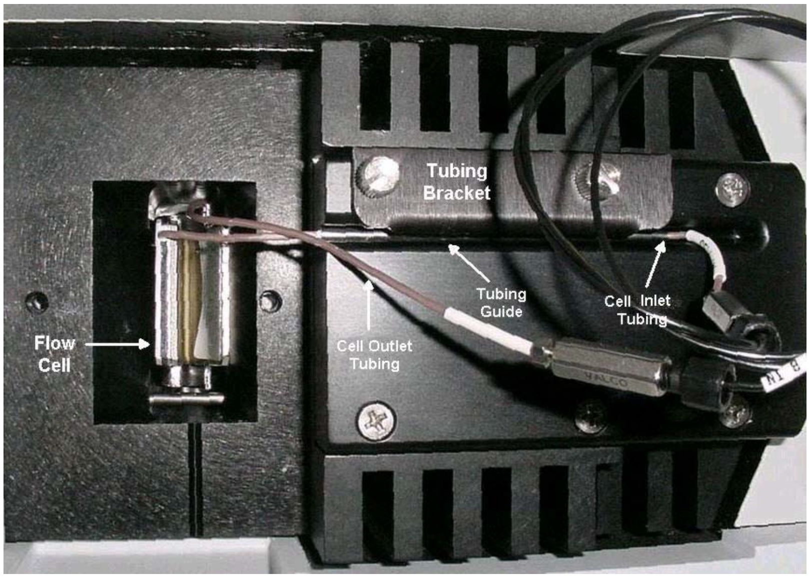

Tubing Bracket
Flow Cell
Cell Outlet Tubing
Tubing Guide
Cell Inlet Tubing

Fig. 30: Removing the Flow Cell from the Optical Bench (Flow cell cover not shown)

Installing a New Flow Cell

Follow the descriptions how to install a flow cell in section 3.6 (→ page 29).

6.2.5 Replacing the Flow Cell Windows and Seals

The description below refers to analytical and semimicro flow cells only. The windows in the semipreparative flow cell cannot be replaced by the user. If cleaning the cell does not eliminate the problems described above, replace the entire cell.

Description	Part No.
Flow cell windows replacement kit, including 2 flow cell windows 2 O-ring seals windows replacement tool	6080.1200

NOTE: Contaminated flow cell windows can significantly increase baseline noise and cause wavelength calibration to fail. If cleaning the cell (→ section 6.2.2, page 86) does not eliminate these problems, replace both silica windows.

1. Remove the flow cell from the optical bench (→ section 6.2.4, page 87) and place the cell on a clean work surface.
2. Put on a pair of latex gloves.

Important: Even minute particles of dust, dirt etc. on the flow cell parts can contaminate the cell and result in excessive baseline noise and drift.

3. Using the windows replacement tool from the replacement kit, remove one of the flow cell retaining nuts from the flow cell (→ Fig. 31). (It does not matter which retaining nut is removed first.)

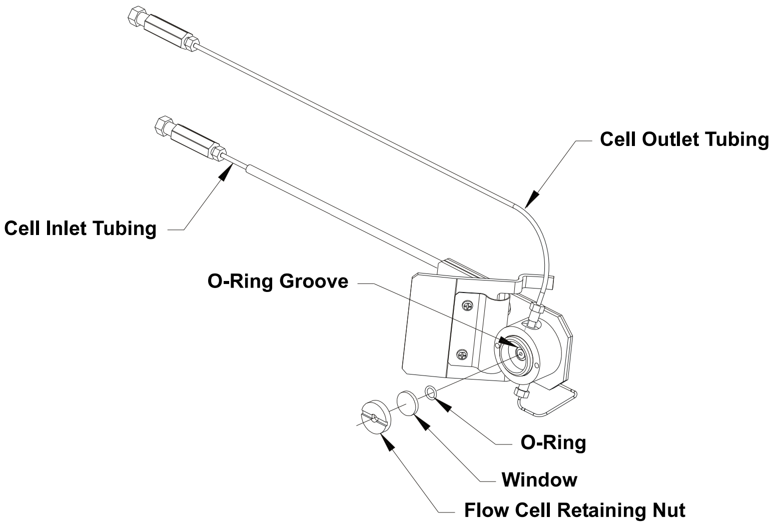


Fig. 31: Replacing the Analytical or Semi-Micro Flow Cell (Light inlet side shown)

4. Remove and discard the old window.

5. Use a needle to carefully remove the seal from the groove. Do not scratch the groove or cell body. Scratches will prevent the new O-ring from sealing properly and cause leakage.

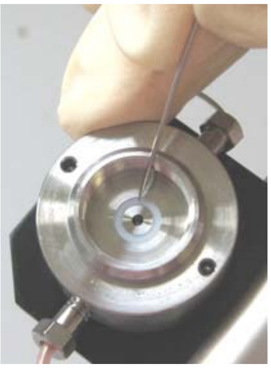


Fig. 32: Removing the seal from the groove

Important: The following factors will prevent the new O-ring from sealing properly and will cause leaks: scratches in the groove, particles of dust or dirt in the groove, and failure to seat the new O-ring in the groove.

6. Check the groove to be sure it does not contain any dust or dirt particles, and then carefully press the new seal into the groove. Do not use a sharp tool as this may damage the seal. Make sure the seal is seated correctly in the groove.
7. Place a new window into the counterbore in the retaining nut. Screw the nut and window into place from below as shown in Fig. 33:

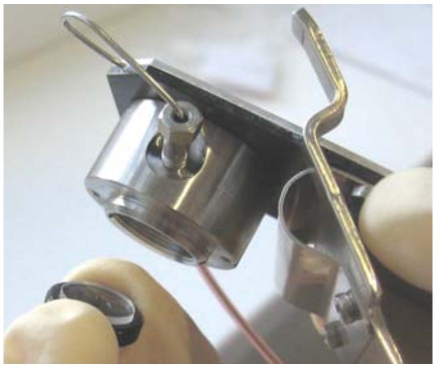


Fig. 33: Placing a new window

8. Using the windows replacement tool, tighten the retaining nut until snug and then turn an additional one-quarter turn.
9. Repeat Step 3 through Step 8 to install the second window.
10. Check the flow cell for leakage (→ page 91).
11. Reinstall the flow cell in the optical bench (→ page 29).

6.2.6 Checking the Flow Cell for Leakage

After you have replaced the flow cell lenses and seals, check the cell for indications for leakage:

1. Connect the flow cell to the pump and install a backpressure capillary on the cell outlet.
2. Pressurize the flow cell as appropriate for the cell type. The backpressure on the cell must not exceed the values specified in the table below:

Flow cell type	**Maximum backpressure**
PEEK analytical or semi-micro cell	21 bar (300 psi)
SST analytical or semi-micro cell	35 bar (500 psi)
Semipreparative cell	100 bar (1450 psi)

3. Inspect the flow cell for indications of leakage. If there is any leakage, tighten the retaining nut(s) with the windows replacement tool until the leak stops. Dry the cell thoroughly with a lint-free, optical-grade tissue.

Important: Do not overtighten the retaining nut, as this may fracture the window.

4. If tightening the retaining nut(s) *does not* stop the leakage, remove the window and inspect the flow cell seal and groove for the cause of the leak. If necessary, clean the groove and reinstall the O-ring (→ section 6.2.5, page 89). Dry the cell thoroughly with a lint-free, optical-grade tissue. If the groove is scratched, the cell must be replaced (→ section 6.2.4, page 87).

6.3 Lamps

6.3.1 Lamp Diagnostics

Chromeleon includes a number of diagnostic functions for monitoring the wellness of your UltiMate 3000 chromatography system. These functions help to prevent problems and ensure reliable operation of each module in the system, as well as to monitor and document service and qualification measures. Section 4.4 describes some of the most frequently used diagnostic procedures.

6.3.2 Replacing the Deuterium Lamp

Description	Part No.
Deuterium lamp	6080.2001

⚠ **Warning**: The deuterium lamp emits UV radiation that is harmful to the eyes. Always turn off the detector power before removing the lamp cover.

⚠ **Avertissement**: La lampe deuterium emettet de rayons ultraviolets, qui sont dangereux pour les yeux. Voulez vous etteindre le detecteur avant d'enlever le couvercle de la lampe.

⚠ **Caution**: The lamp housing and base may be hot to the touch, especially after the lamp has been in operation for a long time. Wait until the lamp has cooled down before continuing.

⚠ **Mise en garde**: La lampe et la base de la lampe peuvent être chaudes au toucher, particulièrement après que la lampe a été allumée pendant longtemps. Attendez que la lampe ait refroidi avant de continuer.

1. Turn off the detector power.
2. Open the detector front panel.

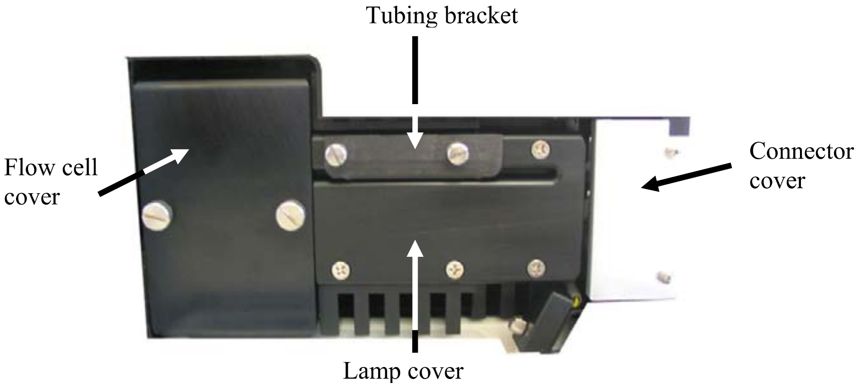


Fig. 34: Covers on PDA inside front panel

3. Remove the flow cell from the optical bench (→ page 87).

   NOTE: It is not required to disconnect the flow cell from the fluidic flow, i.e., step 1 of the description of how to remove the flow cell can be omitted.

4. Move the flow cell tubing out of the way. Remove the four screws holding the lamp cover in place, and then remove the cover and set it aside.
5. Remove the two crosshead screws that hold the metal connector cover on the right in place, and then remove the cover and set it aside.

6. Squeeze the clip on the UV lamp connector (→ Fig. 35) and disconnect it from the 6-pin bulkhead connector on the side wall.

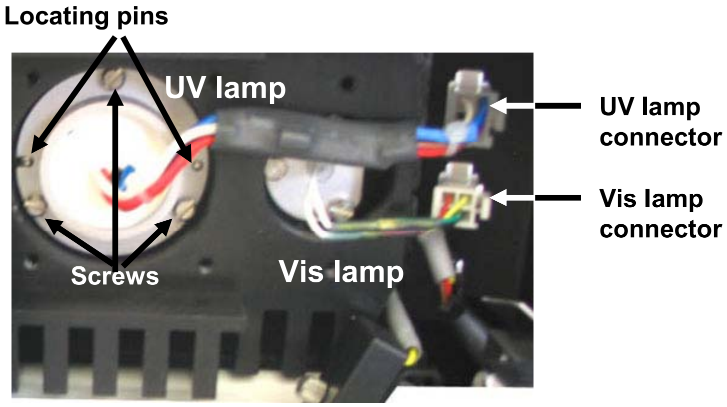


Fig. 35: Lamp Connections

7. Loosen the three screws in the deuterium lamp flange and pull the lamp out of the optical bench.

   NOTE: The screws are captive in the lamp base and do not need to be removed.

8. Inspect the new deuterium lamp for fingerprints and dust. If necessary, clean the lamp with isopropyl alcohol.
9. Line up the flange of the new lamp with the two locating pins and three threaded holes in the optical bench (→ Fig. 35). When the flange is aligned, gently push the lamp into the optical bench.

   NOTE: As opposed to many other optical detectors, the PDA-3000 deuterium lamp forward light beam points away from the flow cell when installed correctly.

10. When the lamp is fully seated, tighten the screws in the flange with a screwdriver.

    Important: To ensure proper performance, the lamp must be fully seated.

11. Squeeze the clip on the UV lamp connector and insert it into the 6-pin bulkhead connector on the side wall.

12. Replace the lamp cover. Route the wires through the guides on the lamp cover inside:

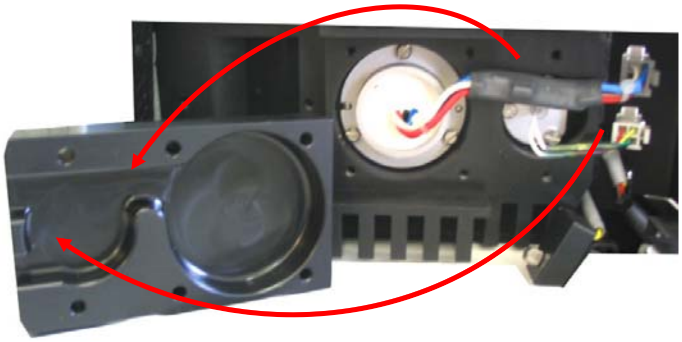


Fig. 36: Replacing the lamp cover

Important: Do not pinch the UV lamp connector wires under the lamp cover.

13. Replace the flow cell and flow cell cover (→ page 31).
14. Close the detector front door.
15. Turn on the detector power.
16. Start the Chromeleon server.
17. Reset the lamp lifetime (→ page 55).
18. Run the wavelength calibration (→ page 56) and wavelength verification test (→ page 57) for the new lamp.

NOTE: Allow the new lamp to run in for 24 hours (minimum) before the first analysis. During this period, strong baseline fluctuations and increased noise may occur.

6.3.3 Replacing the Tungsten Lamp

Description	Part No.
Tungsten lamp	6080.2002

⚠ **Caution**: The lamp housing and base may be hot to the touch, especially after the lamp has been in operation for a long time. Wait until the lamp has cooled down before continuing.

⚠ **Mise en garde**: La lampe et la base de la lampe peuvent être chaudes au toucher, particulièrement après que la lampe a été allumée pendant longtemps. Attendez que la lampe ait refroidi avant de continuer.

1. Turn off the detector power.
2. Open the detector front panel.
3. Remove the flow cell from the optical bench (→ page 87).

   NOTE: It is not required to disconnect the flow cell from the fluidic flow, i.e., step 1 from the description of how to remove the flow cell can be omitted.

4. Move the flow cell tubing out of the way. Remove the four screws holding the lamp cover (→Fig. 34, page 92) in place, and then remove the cover and set it aside.
5. Remove the two crosshead screws that hold the metal connector cover on the right (→Fig. 34, page 92), and then remove the cover and set it aside.
6. Squeeze the clip on the visible lamp connector and disconnect it from the 4-pin bulkhead connector on the side wall (→ Fig. 35, page 93).
7. Use a screwdriver to loosen the tungsten lamp screws, and then pull the mounting assembly away from the optical bench.

   NOTE: The screws are captive in the lamp base and do not need to be removed.

8. Inspect the new tungsten lamp for fingerprints and dust. If necessary, clean the lamp with isopropyl alcohol.
9. Carefully insert the new lamp into the optical bench and tighten the three screws that secure the lamp to the optical bench. (The replacement lamp is pre-aligned in the mount.)

   Important: To ensure proper performance, the lamp must be fully seated.

10. Squeeze the clip on the Vis lamp connector and insert it into the 4-pin bulkhead connector on the side wall. Route the wires through the wire guide in the optical bench (→ Fig. 35, page 93).
11. Replace the lamp cover. Route the wires through the guides on the lamp cover inside (→ Fig. 36 page 94)

Important: Do not pinch the Vis lamp connector wires under the lamp cover.

12. Replace the flow cell and flow cell cover (→ page 31).
13. Close the detector front door.
14. Turn on the detector power.
15. Start the Chromeleon server.
16. Reset the lamp lifetime(→ page 55).

NOTE: Before resuming routine operation, allow 20 to 30 minutes for the lamp to stabilize.

6.4 Replacing the Main Power Fuses

Description	Part No.
Fuse, 4.0 amp fast-blow IEC 127	Fuses kit 6080.9001

⚠ **Danger**: HIGH VOLTAGE—Disconnect the main power cord from its source and also from the rear panel of the PDA-3000.

⚠ **Danger**: HAUTE TENSION—Débranchez le cordon d'alimentation principal de sa source et du panneau arrière du PDA-3000.

1. Before replacing the fuses, turn off the detector. Be sure to disconnect the power cord from its source.

2. The fuse holder is part of the main power receptacle (→ Fig. 37, page 97). Using a small screwdriver (or your fingernails), push the lock upward to release it. (The fuse holder will pop out slightly when the lock is released.) Pull the fuse holder straight out of the compartment.

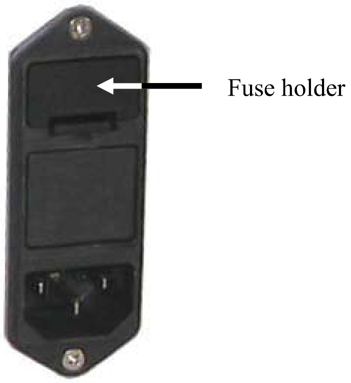


Fig. 37: Main Power Receptacle

3. Replace the two fuses in the holder with new 4.0 amp fast-blow IEC 127 fuses. Dionex recommends always replacing both fuses.

   Important: Do not operate the detector with only one fuse.

4. Reinsert the fuse holder into the compartment. (The fuse holder is keyed to fit only in the correct orientation.) Apply pressure evenly against the holder until the lock is engaged. When correctly installed, the fuse holder is flush against the rear panel.
5. Reconnect the power cord to its source and turn on the detector.

7 Specifications

Noise with Analytical or Semipreparative Cell Installed:	<± 10 µAU at 254 nm, 2-second rise time, 4 nm bandwidth and flowing water at 1.0 mL/min <± 15 µAU at 520 nm, 2-second rise time, 10 nm bandwidth and flowing water at 1.0 mL/min
Noise with Semi-Micro Cell Installed:	<± 15 µAU at 254 nm, 2-second rise time, 4 nm bandwidth and flowing water at 1.0 mL/min <± 15 µAU at 520 nm, 2-second rise time, 10 nm bandwidth and flowing water at 1.0 mL/min
Drift:	<1 mAU/h at 254 nm and 520 nm, and flowing water (after warm-up); typically < 500 µAU/h
Wavelength range:	190 to 800 nm
Wavelength accuracy:	±1 nm, self-calibration with deuterium lines, verification with built-in holmium oxide filter
Resolution:	1 nm
Linearity:	Corr. Coeff. > 99.95 and < 3% RSD up to 1.5 AU
Analytical flow cell:	PEEK cell: 13 µL volume; 10 mm (0.394 in) path length Stainless steel cell: 13 µL volume; 10 mm (0.394 in) path length
Semi-micro flow cell:	PEEKcell: 3.1 µL volume; 9 mm (0.35 in) path length Stainless steel cell: 3.1 µL volume; 9 mm (0.35 in) path length
Semipreparative flow cell:	0.7 µL volume; 0.4 mm (0.02 in) path length
Maximum cell pressure:	Analytical flow cell: < 21 bar (300 psi) (PEEK) < 35 bar (500 psi) (SST) Semi-micro flow cell: < 21 bar (300 psi) (PEEK) < 35 bar (500 psi) (SST) Semipreparative flow cell: < 100 bar (1450 psi)
Optical System	Single-beam, reverse-optics design with concave holographic grating
Photodiode array:	1024 element
Pixel resolution:	0.7 nm

Lamps:	Deuterium lamp (30 W) for ultraviolet spectrum analysis Tungsten lamp (15 W) for visible spectrum analysis
Control:	All parameters and functions controlled by Chromeleon software; the Chromeleon PC and the detector are connected through USB (Universal Serial Bus) 4 LEDs (Power, Run, Deuterium, Tungsten) for status monitoring
GLP features:	In Chromeleon: Support of automatic equipment qualification (AutoQ) and System Wellness monitoring. All system parameters are logged in the audit trail.
Analog outputs:	Four, 0-3 AU, independently selectable, 1000 mV range
Voltage out:	1 V output
Power requirements:	85 to 265 VAC, 47 to 63 Hz auto-sensing; no adjustment necessary) Typical input power: 100 W Maximum line draw: 3.5 A at 110 VAC at power-up
Fuses	Two 4.0 amp fast-blow IEC 127 fuses
Environmental conditions:	Range of use: Indoor use Temperature: 10 to 35°C (50 to 95°F) Air humidity: 80% relative humidity, non-condensing Overvoltage category: II Pollution degree: 2
Dimensions (h × w × d):	19 × 42 × 51 cm (7.5 × 16.5 × 20 in.)
Weight:	19 kg (41.8 lbs)
Decibel level:	<50 dbA during routine operation 56 dbA at power-up

8 Accessories, Spare Parts and Consumables

Accessories, spare parts, and consumables for the detector are always maintained at the latest technical standard. Therefore, part numbers are subject to alteration. However, updated parts will always be compatible with the parts they replace.

8.1.1 Standard Accessories (included in the Ship Kit)

The following accessories are shipped with the detector. (The list is subject to change without notice). Some parts listed below are included in one of the spare part kits. For information about these kits, refer to section 8.1.3 (→ page 102).

Description	Part No.	Quantity in the accessories kit
Standard Accessories for PDA-3000	**5080.8910**	
Screw driver, mini for Relay/TTL connector	6080.1285	1
Fuse, 4A, fast-blow (162VA, 5 x 20 mm)	included in 6080.9001	2
12-pin relay/TTL connector	6080.9010	1
Tubing and Tee piece for waste fluid connection	included in 6000.5001	1 each
Assortment box for accessories	6000.0043	1
USB cable, type A to type B, USB 2.0, 5m	6911.0002	1

8.1.2 Flow Cells

Flow cells must be ordered separately. Cells are shipped with outlet fluidics, backpressure line and capillary to the column compartment and fittings.

Description	Part No.
Analytical PEEK flow cell assembly	6080.0200
Analytical stainless steel flow cell assembly	6080.0210
Semi-micro PEEK flow cell assembly	6080.0220
Semi-micro stainless steel flow cell assembly	6080.0230
Semipreparative PEEK flow cell assembly	6080.0240

8.1.3 Spare Parts and Consumables

Description	**Part No*.**
12-pin relay/TTL connector	6080.9010
Analytical reducing union fitting (PEEK)	6080.2101
Analytical reducing union fitting	6080.2102
Assortment box for accessories	6000.0043
Backpressure line (2 m, 0.25 mm ID) with fitting	6080.2413
Capillary TCC- PDA (0.25 mm ID) stainless steel for analytical cells	6080.2400
Capillary TCC - PDA (0.25 mm ID) PEEK for analytical cells	6080.2421
Capillary TCC - PDA (0.13 mm ID PEEK for semi-micro cells	6080.2422
Capillary TCC- PDA (0.13 mm ID) stainless steel for semi-micro cells	6080.2423
Capillary TCC - PDA (0.5 mm ID) PEEK for semipreparative cells	6080.2424
Cell inlet or outlet tubing PEEK) for semipreparative flow cell	6080.2406
Cell outlet tubing (PEEK) for analytical/semi-micro flow cell	6080.2401
Cell outlet tubing (stainless steel) for analytical/semi-micro flow cell	6080.2403
Deuterium lamp	6080.2001
Female 12-pin connector , RM3.81	6080.9010
Fitting PEEK, 1/16" one-piece fingertight (2 fittings)	6720.0072
Fitting stainless steel, 1/16" screw and ferrule (6 fittings)	6822.0012
Flow cell cover	6080.1210
Flow cell window	6080.1201
Flow cell windows replacement kit, including Windows replacement tool 2 flow cell windows 2 window seals	6080.1200
Fuses Kit, including 4.0 A fast-blow, 5 x 20 mm fuse (10 fuses)	6080.9001
Lamp cover	6080.1211
Outlet fluidics kit (analytical cell), including capillary with tubing and fitting	6080.2410
Outlet fluidics kit (semi-micro cell), including capillary with tubing and fitting	6080.2411
Outlet fluidics kit (semipreparative cell), including capillary with tubing and fitting	6080.2412
Packaging box	5035.8900
Reducing union fitting for analytical/semi-micro flow cell (PEEK)	6080.2101
Reducing union fitting for analytical/semi-micro flow cell (SST)	6080.2102
Screw driver, mini, for Relay/TTL connector	6080.1285
Shipping container (PDA)	5035.8900
Tungsten lamp	6080.2002
Twisted wire assembly, 2 m (8 ft) for relay/TTL connection	6080.9020
Union fitting for semipreparative flow cell	6080.2103
USB cable, type A to type B, USB 2.0, 5m	6911.0002
Waste fluidics kit, including 2 m tubing 5 Tee pieces	6000.5001

* The part number always refers to the packing unit. For more information, please contact your Dionex sales representative.

9 Technical Appendix - Relay/TTL Pin Assignment

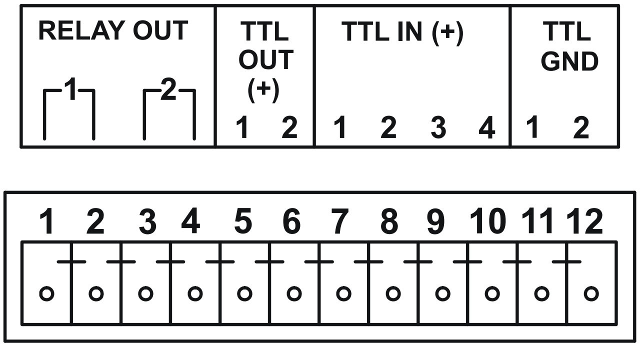


Fig. 38: Relay and TTL Connector

The table below describes the pin assignments for the Relay and TTL connectors. Relays 1 and 2 can be configured in Chromeleon to switch any low-voltage control. The switched current must be less than 200 mA and 42 V peak.

Pin	**Assignment**		**Input Pin Function**
1	Relay 1 output	Active	
2	Relay 1 output	Ground	
3	Relay 2 output	Active	
4	Relay 2 output	Ground	
5	TTL 1 output	Active	
6	TTL 2 output	Active	
7	TTL 1 input	Active	Autozero
8	TTL 2 input	Active	Unassigned - reserved for future use
9	TTL 3 input	Active	UV lamp on/off
10	TTL 4 input	Active	Visible lamp on/off
11	TTL input or output	Ground 1	
12	TTL input or output	Ground 2	

Fig. 39: Relay and TTL: Pin Assignment

10 Index

2D Data - Single Wavelengths..........................10
3D Data..10
  Bunch Width Selection 54
  Reducing Data File Size...............................55
  Step Time Selection 55

A

Absorbance Calculation....................................53
Active Reference Mode 51
  Selecting...51, 76
  Turning off...51
Air Bubbles
  Preventing 74
  Removing...88
Air Conditioning...51
Ambient Temperature Fluctuations 51
Analog Output Connectors 10, 60
Analog Outputs...29
Analytes, unknown 49
Analytical Flow Cell...18
Audit Trail 13, 57, 62
Automated Control 38, 39
Average...44, 46
  Definition 55
  Guidelines for Selecting...............................55

B

Backpressure...34
  Slow Increase 85
  Sudden Increase 85
Backpressure Line...74
Bandwidth...44, 46
  Definition 50
  Setting is too narrow 75
Baseline Drift...46, 51
Baseline Noise 46, 88
  Causes 88
  Effect of Rise Time on… 47
  Reducing 51, 82
Bunch Width...44, 46, 54

C

Cable, USB 28
Calibration, wavelength....................................58
Cells 16, 17, 46, 47
  Air Bubble Prevention 74
  Air Bubble Removal 88
  Backpressure Line..74
  Chemical Compatibility 17, 46, 47
  Cleaning 88
  Heat Exchangers 18, 19
  Installation..33
  Leakage Check 93
  Replacement 89
  Service Procedures.......................................86
  Windows 16
  Windows and Seals Replacement 91
Chlorinated Solvents 17
Chromeleon
  Audit Trail 13, 57, 62
  Commands 44
  Commands Dialog Box................................44
  Control 39
  Detector Installation.....................................32
  Diagnostics 57
  Installation Program.....................................31
  Lamp Diagnostics 93
  Load USB Driver...31
  PDA Control 10, 38
  Server Configuration Program......................31
  Server Monitor...31
Commands Dialog Box 44
Connection
  analog 29
  Power Cord 30
  Relay/TTL Connection 30
  USB 28
Connections 28
Control Commands...44
Control Panel..38

D

Dark Signal...17
Data Collection Rate..................................44, 46
  Definition...48
  Guidelines for Selecting 48, 49
  Relationship to Step.....................................54
Data Points 48, 54
Deuterium Lamp...10, 16
  Age 45
  Calibration 78
  Failure...13
  Intensity 57
  Lamp does not light 72
  On/Off Control 45
  Output Waning 74
  Replacing...94
  Time to Stabilize..............................37, 73, 76
Deuterium LED 71, 72, 73
Diagnostics 93
Disk Space..46, 49
Drain...35
Drain Tube..35

E

Electronics 85

F

Fan 68
Ferrule Fittings 73
Filter Paddle 16, 17, 75, 78
Flashing LED 71
Flow Cells 17
  Air Bubbles 88
  Chemical Compatibility 17, 47
  Cleaning 88
  Installation 33
  Leakage Check 93
  Leaks 87
  Maintenance 86
  Material 47
  Replacement 89
  Windows and Seals Replacement 91
Found New Hardware Wizard 32
Front Panel 15
Front Panel - LEDs 13
Fuse Holder 99
Fuses 99

G

Gradient Analyses 77
Gradient Applications
  Gradient Elution 23
  Negative Absorbance 56
  Reference Wavelength Selection 51
Grating (optical) 16, 17

H

Heat Exchanger Volume 18, 19, 20
Heat Exchangers 18, 19
Holmium Oxide Filter 10, 17
Hub, USB 28

I

Inside Front Panel 15
Installation
  Flow Cell 33
  in Chromeleon 31
  Rear Panel Connections 28
  Relay/TTL 105
  Site Requirements 25
  Software 28
  UltiMate 3000 System 26
  Waste Tubing 35
Integrator 29, 81
Intensity (Lamp) 57
Isocratic Analyses 51, 77

L

Lamp Connections 95
Lamp Diagnostics 93
Lamp Intensity 57
Lamps 10
  Calibration 78
  Failure 13, 14
  Installation Procedures 94, 97
  Replacement 93
Leak Sensor 22
Leak Sensor Error 68
Leak Tray 85
Leaks
  Flow Cell 87
  Leak Sensor 22
  Leak Sensor Error 68
  Symptoms 85
LED Flashing 13, 14
Lenses 16
Liquid Lines - Maintenance 85, 86, 87

M

Maintenance 85
Mobile Phases
  Concentration Guidelines 17, 23, 47
  Degassing 23, 74
  Lamp Stability after Changing 73, 76
  Preparation 73
  Reservoir Material 24
  Retention Time 23
  Solvent Quality 23
  Temperature Stabilization 18, 19

N

Negative Absorbance 44, 46, 56, 76
Normal Phase Solvents 17
NOTES in manual 6

O

Offset Level 44
Operating Instructions 4
Operating Parameters ... 46, 48, 49, 50, 51, 52, 54, 55, 56
Operation
  Safety Precautions 6
  Safety Symbols 5
Optical Bench 68
Optical System
  Components 16
  Illustration 16

P

Parts Replacement 85, 103
Passivation 86
PDA 3000 Applications 37
Peak Height...47
Peak Identification 12, 49
Peak Purity...10, 12, 49
Peak Resolution 46
Peaks
  Asymmetrical 47
  Co-eluting 48
  Improving height..81
  Improving shape...82
Performance Optimization..46, 48, 49, 50, 51, 52, 54, 55, 56
Performance Optimization Time........................47
PGM Files...39, 80
PGM Wizard 39
Photodiode Array..................................10, 16, 17
Photodiode Array Detection 11
Pin Assignments (Relay/TTL) 105
Post-Column Reagents................................74, 81
Power Cord 30, 99
Power LED 71
Power Switch 14, 31
Power Up Diagnostic Test 57
Problems 61
Pumping System Requirements 23

R

Rear Panel..21
Rear Panel Connections 28
Recorder...29, 81
Reference Bandwidth..................................44, 46
  Definition 52
  Guidelines for Selecting...................50, 51, 52
Reference Intensity 51
Reference Modes 51
  Active Reference Mode 51
  Active Reference Wavelength.......................76
Reference Wavelength....................44, 46, 51, 52
  Definition 51
  Guidelines for Selecting...............................51
  Inappropriate Setting...................................75
Relay and TTL Connector 105
Relay Control..30, 105
Rise Time..44, 46
  Correlation with Data Collection Rate 49
  Definition 47
  Effect on Baseline Noise..............................47
  Guidelines for Selecting..........................47, 49
  Inappropriate Setting...................................75
RMA Number 85
Run LED...13, 71

S

Safety Icons
  Caution 5
  Danger 5
  Important 5
  Warning 5
Safety Information... 5
Safety Precautions 6
Safety Symbols.. 5
Sample Bandwidth 50
Sample Intensity 51
Sample Rate (see Data Collection Rate)............ 48
Sample Wavelength.............................. 49, 50, 51
Sample Wavelength... 49
Semi-Micro Flow Cell 19
Semipreparative Flow Cell 20
Sensitivity.. 46
Separator Column 85
Service Procedures
  Cell Cleaning 88
  Deuterium Lamp 94
  Flow Cell 86
  Fuse Replacement... 99
  Lamps 93
  Removing trapped air from cell 88
  Tungsten Lamp Replacement 97
Shutdown Procedure... 85
Signal Intensity.. 51
Slit (optical)... 16, 17
Source Lens 16
Spare Parts.. 103
Spectra not collected... 80
Spectral Library 10, 12
Spectrograph Lens 16, 17
Spectrum... 16, 17
Spectrum - Improving Resolution 81
Step... 45, 46
  Definition.. 54
  Guidelines for Selecting 54
  Selection by Software 54, 68
System Wellness.. 93

T

Technical Support.. 37
Temperature (ambient) 51
Timebase 73
Troubleshooting... 61
  Baseline Drift.. 76
  Baseline Noise 73
  Diagnostic Tests 57
  Elution time is slow 74
  Filter Paddle Malfunction 75, 78
  Lamp does not light 72
  Lamp Intensity.. 77
  PDA-3000 does not respond......................... 72
  Peak shape is poor 82

  Peak size is wrong..81
  Spectra not collected....................................80
  Spectral resolution is poor 81
  USB communication problem.......................83
  Wavelength calibration fails 78
  Wavelength verification fails........................80
TTL Control..30, 105
TTL Input Mode 45
Tubing Connections.........................24, 85, 86, 87
Tungsten Lamp 10, 16
  Age...45
  Calibration 78
  Failure 14
  Intensity 57
  Lamp does not light 72
  On/Off Control..45
  Output Waning..74
  Replacing 97
  Time to Stabilize 37, 73, 76
Tungsten LED.......................................71, 72, 73

U

Unpacking Instructions......................................25
USB 10, 28
  Configuration File 73, 83
  Connection to Hub....................................... 28
USB Cable.. 28
USB Communication Problem.......................... 83
UV Wavelengths 16

V

Ventilation.. 76
Verification, wavelength 59
Visible Focus Lens 16
Visible Wavelengths... 16

W

Warranty, Voiding.. 85
Waste Tubing 35
Wavelength.. 10, 45
Wavelength Calibration......................... 45, 58, 80
Wavelength Verification 17, 45, 59
Wavelengths, single.................................... 49, 51